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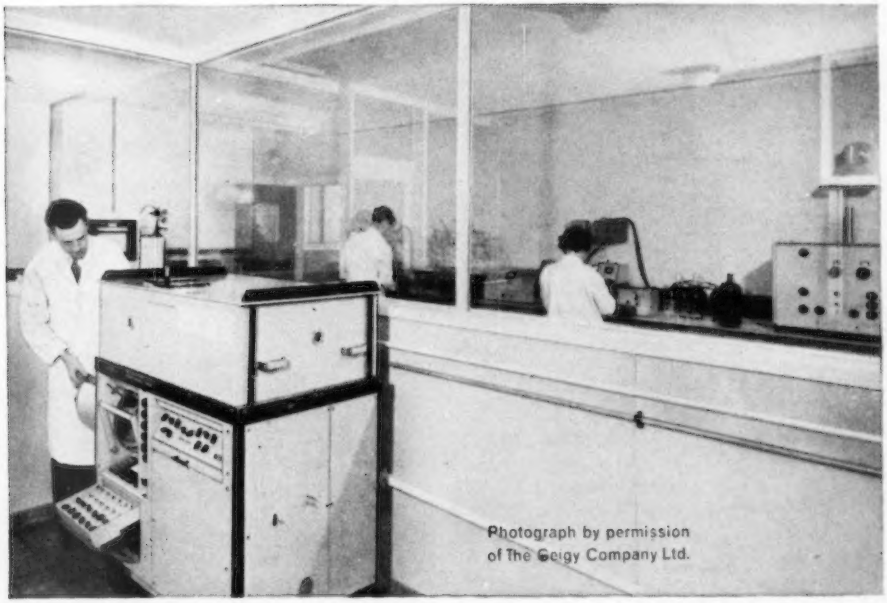

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ANALYTICAL ABSTRACTS

1.—GENERAL ANALYTICAL CHEMISTRY

General reviews of progress, reagents and methods of general application.

2732. **Recommendations for terminology to be used with precision balances.** International Union of Pure and Applied Chemistry. Commission on Microchemical Techniques. *Pure and Appl. Chem.*, 1960, **1** (1), 171–175.—Definitions of the usual terms are given in English, French and German.
G. J. HUNTER

2733. **Terminology for scales of working in microchemical analysis.** International Union of Pure and Applied Chemistry. Commission on Microchemical Techniques. *Pure and Appl. Chem.*, 1960, **1** (1), 169–170.—The classification is recommended by the Committee on Microchemical Techniques of the I.U.P.A.C. and is regarded as particularly important with respect to methods involving < 1 mg of sample. G. J. HUNTER

2734. **Report on the standardisation of pH and related terminology.** International Union of Pure and Applied Chemistry. Commissions on Electrochemical Data and Physico-chemical Symbols and Terminology. *Pure and Appl. Chem.*, 1960, **1** (1), 163–168.—The extent and the nature of the standardisation of pH are discussed. Tables of the values of pH over a range of temperatures from 0° to 95° of K H tartrate (satd. at 25°), 0·05M-K H phthalate, 0·025M-KH_2PO_4 - 0·025M-Na_2HPO_4, and 0·01M-$Na_2B_4O_7$ are given. G. J. HUNTER

2735. **Radioactive isotopes in medicine: a review.** C. J. Hayter (Isotope Lab., St. Thomas's Hospital, London, S.E.1). *J. Clin. Path.*, 1960, **13** (5), 369–390.—After a brief review of the counting techniques suitable for use in a hospital laboratory, some of the diagnostic uses of radio-isotopes are discussed. These are divided into haematological and biochemical tests, with a further sub-section on tests of thyroid function. R. A. BRENAN

2736. **Characteristic constants of 2,2′,4′-trihydroxyazobenzene-5-sulphonic acid, a reagent for spectrophotometric analysis.** M. H. Fletcher (U.S. Geol. Survey, Washington, D.C.). *Anal. Chem.*, 1960, **32** (13), 1822–1827.—This dye is promising as a reagent for the determination of Zr. The acid was pptd. from the commercial dye and recrystallised from dil. HCl as a dark-red, hygroscopic crystalline material which could be dried to the monohydrate at 150° and to the anhydrous acid at 290°. The sulphonic acid group and the three hydroxyl groups all show acid characteristics. The spectra of a soln. of the dye were plotted for a wide range of acidities and the dissociation constants were determined. The results presented serve to define the absorption spectrum at acidities between 10·35N-HCl and pH 11·9. G. BURGER

2737. **The acid - base properties of Solochrome violet RS. I.** H. Khalifa and S. W. Bishara (Nat. Res. Centre, Dokki, Cairo, Egypt). *Z. anal. Chem.*, 1960, **178** (3), 184–193 (in English).—Solochrome violet RS (C.I. Mordant Violet 5) has been compared with its nitro-analogue Fast Grey RA; unlike the latter compound it is specific for V and Mo. Its properties as an acid - base indicator were examined by titration of HCl, HNO_3, H_3PO_4, H_2SO_4, acetic, oxalic and tartaric acids with NaOH. When alkali was used as the titrant, sharp end-points were obtained for most acids of $\simeq$ 0·1N concn. Potentiometric titration of the dye in acid form with NaOH yielded data from which pK values of 4·35, 7·4 and 9·35 were obtained.
P. D. PARR-RICHARD

2738. **Phloroglucinol—an indicator for mercurimetry.** B. D. Berezin and L. G. Pogodina (Ivanovo Chem.-Tech. Inst.). *Zavod. Lab.*, 1960, **26** (12), 1347–1351.—The volumetric determination of Cl^- in strongly acid soln. by means of $Hg(NO_3)_2$, with phloroglucinol as indicator, does not require the presence of glucose, urea or other organic substance, as has been stated by Mavlyanov (*Zhur. Prikl. Khim.*, 1937, **10**, 1162). The optimum conditions for titration are 0·1N-Hg^{2+}, 0·1N-Cl^- and 0·1 to 0·2 ml of 0·1 % phloroglucinol soln. in a final vol. of 30 to 40 ml at a pH of $\simeq$ 2.
G. S. SMITH

2739. **Stilbazo as metallochromic indicator. Preliminary communication.** J. Ditz (Res. Inst. of Inorg. Chem., Ústí nad Labem, Czechoslovakia). *Z. anal. Chem.*, 1961, **178** (4), 274–277 (in German).—Stilbazo R [diammonium salt of 4,4′-di-(3,4-dihydroxyphenylazo)stilbene-2,2′-disulphonic acid] (**I**) has properties of an acid - base indicator, its colour changes being from yellow to red - violet, then to red - orange and finally to violet and blue. With metal ions **I** forms coloured complexes and the results quoted show that the end-point in the EDTA titration of Th^{4+} or Bi^{3+} with **I** as indicator is very sharp. *Procedure for Bi or Th*—To the sample soln. are added 10 ml of acetate buffer (pH 3·8, prep. described) and 5 drops of 0·5% aq. **I**, and the soln. is diluted to 100 ml and titrated with 0·05M-EDTA (disodium salt). The end-point is marked by a colour change from violet to yellow. The error is $\pm$ 0·5% for 80 mg of Th and $\pm$ 0·7% for 50 mg of Bi in 100 ml of soln. B. B. BAUMINGER

2740. **Contributions to the problem of determination of the equivalence-point. I. A new method of calculation of the equivalence-point in linear titrations.** C. Liteanu and D. Cörmös (Univ. Babes-Bolyai, Cluj, Romania). *Talanta*, 1960, **7** (1–2), 18–24 (in French).—Linear titrations (conductimetric, amperometric, heterometric, etc.) may be best evaluated by calculating the slopes of the two lines obtained on plotting vol. of titrant against instrument response by the method of least squares, and using these to calculate the true point of intersection.

II. A new method for the determination of the real asymmetry coefficient in potentiometric titrations. C. Liteanu and D. Cörmös. *Ibid.*, 1960, **7** (1–2), 25–31 (in French).—After linearisation of the titration curve by the procedure of Gran (*Acta Chem. Scand.*, 1950, **4**, 559) (plotting of dV/dE), the angular coeff. of the two lines before and after the end-point are calculated by the method of least squares and these are used, together with titration results obtained some distance from the end-point, to calculate the real coeff. of asymmetry.

III. A new method for calculation of the equivalence-point in potentiometric titrations. C. Liteanu and D. Cörmös. *Ibid.*, 1960, **7** (1–2), 32–40 (in French).—The point of intersection of the two straight lines obtained by linearising the potentiometric titration curve, together with the asymmetry coeff., are used to calculate the true equivalence-point. T. R. ANDREW

2741. **Fundamental principles of titrations with potassium bromate.** E. Schulek, K. Burger and J. Laszlovszky (Inst. of Inorg. Anal. Chem., L. Eötvös Univ., Budapest). *Talanta*, 1960, **7** (1–2), 51–55 (in English).—It is shown that the active agents in titrations with $KBrO_3$ are Br, BrCl, or BrCl plus Cl, according to the halide concn. In the presence of excess of Br^-, elementary Br is formed; with a limited amount of Br^-, BrCl is produced; and in the absence of Br^- and the presence of Cl^-, BrCl and Cl are formed.
T. R. ANDREW

2742. **Influence of acids and salts on the use of chromous chloride as a reducing titrant.** W. U. Malik and K. M. Abubacker (Muslim Univ., Aligarh, India). *Anal. Chim. Acta*, 1960, **23** (6), 518–523 (in English).—The potentiometric titration of $CuSO_4$ with 0·27N-$CrCl_2$ is conducted in the presence of various acids and salts. Satisfactory titration curves are obtained only in acid soln. with a suitable concn. of Cl^-. High temp. are undesirable.
H. N. S.

2743. **Study of some solvents as media for the high-frequency titration of weak acids.** E. L. Grove and W. S. Jeffery (School of Chem., Alabama Univ., U.S.A.). *Talanta*, 1960, **7** (1–2), 56–64 (in English).—The behaviour of methyl cyanide, dimethylformamide, ethylenediamine, pyridine, n-butylamine and piperidine as solvents in the high-frequency titration of weak acids has been studied. Solvents with high dielectric constants and very weak basicities are recommended for high-frequency and conductimetric titrations. T. R. ANDREW

2744. **Thermal stability of analytical standards. IX.** C. Duval and C. Wadier (Lab. de Rech. Microanal., Paris, France). *Anal. Chim. Acta*, 1960, **23** (6), 541–544 (in French).—Thermolysis curves of the following substances are recorded, together with notes on their i.r. absorption spectra—Cd acetate dihydrate, KBr, $La_2(SO_4)_3.8H_2O$, $CoSO_4.7H_2O$, $MgSO_4.7H_2O$, K_2SO_4, guanidine carbonate, phthalic anhydride, phthalic acid, Li citrate, malonic acid and succinic acid. H. N. S.

See also Abstracts—**2768**, Trialkyl thiophosphates for extraction of Ag and Hg. **2781**, 1-Phenyltetrazoline-5-thione as reagent. **2882**, Preparation of furil α-dioxime. **2910**, Use of BrCl as reagent.

2.—INORGANIC ANALYSIS

General, determination of elements (arranged in the order of the Periodic Table), analysis of minerals and inorganic industrial products.

2745. **Methods for analysis of impurities in solids.** F. Trendelenburg (Forschungslab. der Siemens-Schuckertwerke, Erlangen, Germany). *J. Sci. Ind. Res., India, A*, 1960, **19** (11), 542–549.—The following methods for the determination of trace amounts of elements are reviewed critically, with indications of their scope and limitations and of the degree of efficiency to which they have been developed, *viz*, emission spectroscopy (*e.g.*, for checking the purification of metals by zone melting), flame photometry (*e.g.*, for the determination of 10^{-9} g of Na or 10^{-7} g of K per ml, and 10^{-6} g of Na in high-purity aluminium), i. r. spectrometry, mass spectrometry (the limiting sensitivity is 10^{-10} to 10^{-13} mole of gas per g of solid), gas chromatography (the sensitivity is $\simeq 1$ in 10^6), colorimetry ($\simeq 14$ elements can now be determined in minimum concn. of $\simeq 0{\cdot}01$ μg per ml), polarography (this permits 10^{-5}% of an impurity to be directly determined in a metal), nuclear magnetic resonance (the sensitivity of detection is 10^{16} protons per cu. cm and 5×10^{-4} % of H_2O in ^3H_2O), radiochemical activation analysis (applicable to $\simeq 75$ elements, of which $\simeq 68$ can be detected to within 10^{-7} g), and the radioactive-tracer method (*e.g.*, the determination of contaminants during the purification of arsenic and antimony). W. J. BAKER

2746. **Thermodynamic method for determination of the conditions for analysis of gases in metals and its use for the development of the technique of analysis.** Yu. A. Klyachko and E. M. Chistyakova (I. P. Bardin Central Sci. Res. Inst. of Ferrous Metallurgy). *Zavod. Lab.*, 1960, **26** (12), 1335–1338.—From thermodynamic data, the change of free energy in reactions such as the reduction of oxides by carbon and the formation of carbides during dissolution of the metal in iron, temperature - composition diagrams of alloys, etc., it is possible to derive suitable conditions for the determination of gases in metals. In the specific case of manganese it is shown that the addition of Cu to decrease the concn. of Mn to $\not> 30$ % is desirable and that the extraction of gases can be carried out at $\not> 1100°$.
G. S. SMITH

2747. **Sequential ion-exchange separation scheme for the identification of metallic radio-elements.** W. J. Blaedel, E. D. Olsen and R. F. Buchanan (Chem. Dept., Univ. of Wisconsin, Madison). *Anal. Chem.*, 1960, **32** (13), 1866–1873.—A procedure is described by which, in trace amounts, radio-elements are separated into 6 groups. The sample is dissolved in citric acid soln. - aq. NH_3 at pH 3, treated with a stream of Cl and adsorbed on a column of Dowex 50W-X8 resin pre-treated with Cl. The elements are then eluted with a series of complex-forming eluents at controlled pH and ionic strength, and the eluates are continuously monitored for radioactivity. The elements of each group are obtained in 15 to 30 ml of soln. containing only ammonium salts, organic acids or HCl. Each of 36 elements falls mainly into its own group, with < 1% falling into any other group. Recoveries of 31 elements were >90%; Pd, Au, Hg and Ag must be absent.
R. E. E.

2748. **8-Hydroxyquinoline precipitation of the elements.** J. I. Hoffman (Nat. Bur. of Stand., Washington, D.C., U.S.A.). *Chemist Analyst*, 1960, **49** (4), 126 (in English).—The elements are divided into four groups—those completely or partially pptd. from acetate-buffered soln. (pH $\simeq$ 5·7) and those completely or partially pptd. from ammoniacal soln. (pH > 7·5). W. T. CARTER

2749. **Procedure for determining the molar extinction coefficients of metal dithizonates.** H. Irving and R. S. Ramakrishna (Inorg. Chem. Lab., South Parks Rd., Oxford, England). *Analyst*, 1960, **85**, 860–867.—Methods for evaluating the mol. extinction coeff. of metal complexes with dithizone are reviewed and a method of general applicability is proposed and illustrated by the determination of the mol. extinction coeff. of the silver and mercury dithizonates. Portions (10 ml) of dithizone soln. of different concn. in CCl_4 are shaken mechanically with 10 ml of the metal soln. The organic phase is freed from water by centrifuging, and the extinctions are measured at the wavelength of max. absorption of the metal complex and of the dithizone. A 5-ml portion is then shaken with 5 ml of a reversion reagent (*i.e.*, a reagent that removes the metal from the complex), and the extinction of the organic phase is measured at 620 mμ and 450 mμ. The reversion reagent for Hg is an aq. soln. of KI containing K H phthalate and that for Ag a soln. of KI in 0·25N-H_2SO_4. Results for the Ag and Hg systems are given and it is shown that the method should be equally applicable to other metal - reagent systems. A. O. JONES

2750. **Paper-chromatographic and paper-electrophoretic separation of elements of the reduction group.** E. Blasius and M. Fischer (Inorg. Chem. Inst., Tech. Univ., Berlin). *Z. anal. Chem.*, 1960, **178** (1), 28–33 (in German).—The techniques of paper chromatography and electrophoresis are combined for the detection and separation of the mixtures Pd - Au - Se - Te, Pd - Pt - Au - Te, and Pt - Au - Se - Te. Butanol - 3N-HCl (7:3) is a suitable developer (ascending technique) for Au - Pt - Se - Te and Au - Pt - Pd - Te, and butanol - 10N-HNO_3 (7:3) for Au - Pd - Se - Te. The chromatogram is sprayed with a reducing soln. of HCl - NaI - $SnCl_2$; the resulting spots of the elements show characteristic colours (Pd, brown; Pt, yellow; Au, violet - black; Se, orange - yellow; and Te, brown). This paper-chromatographic method can be applied to naturally occurring gold tellurides, *e.g.*, nagyagite and sylvanite. The sample (10 mg) is dissolved in 1 to 2 ml of aqua regia, and the soln. is evaporated and diluted with water to 1 ml; 0·01 ml of this soln. is spotted on to filter-paper. The paper is then sprayed with rhodizonic acid (for the detection of Pb) and a soln. of HCl - NaI - $SnCl_2$ (for Au and Te). Paper electrophoresis has been applied to the separation of all 5 elements. With a p.d. of 110 to 130 V and a buffer of pH 2·7 [N-HCl - N-Na acetate (1:1)] the separation is achieved in 1 to 2 hr. B. B. BAUMINGER

2751. **Spectrographic analysis of brass by high-frequency discharge excitation.** E. S. Kudelya (Inst. of Metalloceramics and Special Alloys, Acad. Sci., UkrSSR). *Zavod. Lab.*, 1960, **26** (12), 1374–1375.—The determination of Zn, Pb, Si, Sn, Mn and Fe in brass by means of a high-frequency spark from a Tesla transformer is described. G. S. SMITH

2752. **Chronopotentiometric analysis. I. Behaviour of copper, lead, cadmium and bismuth ions in various supporting electrolytes.** Masayoshi Ishibashi, Taitiro Fujinaga, Toyoshi Nagai and Susumu Kakumoto (Chem. Dept., Fac. of Sci., Kyoto Univ., Sakyo-ku). *J. Chem. Soc. Japan, Pure Chem. Sect.*, 1960, **81** (1), 88–92.—The quarter-wave potential and the transition-time constant of these ions in 0·1N- and N-HCl, HNO_3, H_2SO_4 and $HClO_4$ are compared with the half-wave potential and the diffusion-current constant, respectively. Good correspondence was observed with these ions except those whose electrode reaction is influenced by gelatin. The chronopotentiogram has no max., but the reproducibility of the wave shape and of the transition-time constant is sometimes affected by a surfactant such as polyacrylamide (**I**).

II. Effect of surface-active substances on the chronopotentiograms of copper, lead, cadmium and bismuth ions in various supporting electrolytes. Toyoshi Nagai. *Ibid.*, 1960, **81** (1), 93–94.—The chronopotentiograms of these ions are not affected by **I** or gelatin (0·001%) in HCl, KCl, NH_4Cl, aq. NH_3 or NaOH soln. In $HClO_4$, H_2SO_4 and HNO_3, the quarter-wave potential shifts towards the negative region and the transition-time constant decreases. While the corresponding amalgam oxidation curves are little affected by surfactants, the reduction at the electrode appears to be influenced by gelatin. A similar relationship was observed with Bi in 0·1N-EDTA soln. Reproducibility of the wave shape and of the transition-time constant of Cu in HNO_3 is much improved by **I** (0·001%).

III. Determination of copper, lead, cadmium and bismuth ions in various supporting electrolytes. Toyoshi Nagai. *Ibid.*, 1960, **81** (2), 250–252.—The relationship between the concn. of these ions and the square root of the transition time was examined in HNO_3, HCl, KCl, NaOH, NH_4Cl and aq. NH_3 soln. Suitable electrolytes are M-NH_4Cl - aq. NH_3 soln., N-HNO_3 containing 0·001% of **I** for Cu, 0·1N-NaOH and 0·1M-KCl for Pb, 0·1M-KCl and 0·1M-NH_4Cl - aq. NH_3 soln. for Cd, and N-HNO_3 and N-H_2SO_4 for Bi. The error is $<3\%$ for 2×10^{-4} to 1×10^{-3}M and $\simeq 3\%$ for 2×10^{-5} to 1×10^{-4}M soln.

IV. Behaviour of the zinc ion in various electrolytes. Toyoshi Nagai. *Ibid.*, 1960, **81** (2), 252–254.—The quarter-wave potential of Zn in 0·1M-KCl, 0·1M-KSCN, 0·1M-NH_4Cl - aq. NH_3 and 0·1M-NaOH agrees with each half-wave potential. Both the shape and the transition-time constant are affected by gelatin, even in those electrolytes that form insol. salts with Hg. For the determination of 10^{-3} to 2×10^{-4}M-Zn, 0·1M-NH_4Cl containing aq. NH_3 is the best supporting electrolyte, the deviation being $<1\%$.

V. Behaviour of iron(III) towards the platinum electrode. Toyoshi Nagai. *Ibid.*, 1960, **81** (2), 254–256.—In 0·5N-HCl, the quarter-wave potential is + 0·46 V *vs.* the S.C.E. and the characteristic potential of the re-oxidation wave is more positive by 15 mV. The transition-time constant is 214 amp. per cm per sec.$^{0.5}$ per mole and agrees with the calculated value when it is assumed that the reaction involves the transfer of one electron. A linear relationship holds between the square root of the constant and the concn. of Fe for 4×10^{-4} to 10^{-2}M, the error being $< 3\%$. In a more dilute soln. the wave is less reproducible. Dissolved O gives a reduction wave at + 0·2 V and vitiates the determination.

VI. Investigation of the chronopotentiometry of binary mixtures and the determination of a small

amount of lead and cadmium in zinc oxide. Toyoshi Nagai. *Ibid.*, 1960, **81** (2), 256–259.—The mutual influence of Pb and Cd was examined in 0·1M-KCl. The ratio of the transition times is constant irrespective of the current density for a given concn. of each ion. For a given concn. of Cd ($\simeq 10^{-4}$M) and a given current density ($\simeq 10^{-5}$ amp. per sec.), the transition time increases linearly with increase in concn. of Pb ($\simeq 10^{-4}$M), but the relationship between the concn. of Cd and the transition time is parabolic. For the determination of Cd, the molar concn. of Pb must be $<$ 15 times that of Cd. The results agree well with those obtained by polarography.

VII. Behaviour of cobalt and nickel in various electrolytes. Masayoshi Ishibashi, Taitiro Fujinaga, Toyoshi Nagai and Susumu Okamoto. *Ibid.*, 1960, **81** (5), 744–747.—The quarter-wave potentials of Ni and Co in 0·1M- and M-NH_4Cl soln. containing aq. NH_3, 0·1M-KCl and M-KSCN agree with the half-wave values. Gelatin causes a shift in potential towards the negative by 80 mV, but **I** has little influence. The determination of Ni and Co is carried out in 0·1M-NH_4Cl containing aq. NH_3, the error being $<$ 1% for 2×10^{-4} to 10^{-3} moles of Ni, and $\simeq$ 3% for 4×10^{-4} to 10^{-3} moles of Co.

K. Saito

2753. **Method for the separation of titanium, zirconium, iron and aluminium from one another and for their subsequent determination.** T. J. Murphy, W. S. Clabaugh and R. Gilchrist. *J. Res. Nat. Bur. Stand., A*, 1960, **64** (6), 535–542.—The new procedure described is applicable to barium titanate ceramics. The four elements are first collectively pptd. from an HCl soln. of the sample with a large excess of 8-hydroxyquinoline at pH 5, and the ppt. is dissolved in warm dil. HCl containing citric acid (to ensure subsequent masking of Ti and Zr). From this soln. the Fe is pptd., at 60° and pH 1·5, with a slight excess of 1-nitroso-2-naphthol in the presence of $\simeq$ 20% (v/v) of ethanol; the ppt. is ignited at 900° and weighed as Fe_2O_3. The Ti and Zr in the filtrate are then co-pptd. with cupferron at 60°, leaving in soln. only Al, which is determined as Al_2O_3 after pptn. as Al cupferrate at 0° and pH 4. The mixed cupferrates of Ti and Zr are ignited (at $\not>$ 450°) to the oxides, which are then dissolved in dil. H_2SO_4, and solid EDTA is added ($\simeq$ 0·5 g in excess) to mask Zr. The Ti is pptd., under carefully controlled conditions at pH 2 to 4·5, with 8-hydroxyquinoline and is weighed finally as TiO_2 (any SiO_2-free residue from the dissolution of the oxides is included). The Zr in the filtrate (made acid with HCl) is pptd. at $\simeq$ 0° with cupferron ($\simeq$ 1 g in excess) and is weighed as ZrO_2. The errors are usually within $\pm$0·2 mg for concn. of 0·2 to 0·5 g of TiO_2, $\not>$ 0·25 g of ZrO_2 and $\not>$ 0·05 g each of Fe_2O_3 and Al_2O_3 (eight analyses of synthetic mixtures).

W. J. Baker

2754. **Separation and successive determination of iron, titanium and aluminium by means of diantipyrinylmethane.** V. P. Zhivopistsev and A. A. Minin (Perm State Univ.). *Zavod. Lab.*, 1960, **26** (12), 1346–1347.—The sample soln. containing Fe, Ti and Al is adjusted to 5 to 6N in HCl and the Fe is extracted by shaking with 10 to 25 ml of $CHCl_3$ in the presence of 0·5 to 0·7 g of diantipyrinylmethane (**I**). When the content of Fe is low, the extinction (blue filter) of this extract can be used to give the content. One or two further extractions with 10 to 15 ml of $CHCl_3$ and 0·2 to 0·3 g of **I** are made. They are rejected if the content of Fe is low, but combined with the first extract if the content is high. In this case the combined extracts are washed with water and the Fe in the aq. extracts is determined gravimetrically as Fe_2O_3 after pptn. as $Fe(OH)_3$. To determine Ti in low concn., the Fe-free acid soln. or an aliquot thereof is treated with sufficient **I** to give a 2% soln. and the Ti is determined colorimetrically. If the content of Ti is high, the soln. is extracted with 10 to 15 ml of $CHCl_3$ in the presence of 1 g of **I** and 20 ml of 20% NH_4SCN soln. and then with 10 ml of $CHCl_3$. The combined extracts are mixed with an equal vol. of ethanol and treated with conc. aq. NH_3 until the odour of NH_3 persists. The soln. is heated gently until it is decolorised, and the ppt. is collected and ignited to TiO_2. The Al, which may be accompanied by Mn, Cr, Ni, alkaline-earth metals and alkali metals, which do not react with the reagent, is determined in the residual soln. No interference is caused by the presence of F^-, PO_4^{3-} and small amounts of SiO_2.

G. S. Smith

2755. **The chelatometric determination of heavy metals with nitrilotriacetic acid. I. Direct titration of nickel, copper, zinc and cadmium.** Gen-Ichiro Numajiri and Mutuo Kodama (Fac. of Arts and Sci., Ibaraki Univ., Mito). *J. Chem. Soc. Japan, Pure Chem. Sect.*, 1960, **81** (3), 454–457.—Nickel, Cu and Zn are titrated with Na nitrilotriacetate (**I**) in ammoniacal soln. of pH 8·2 to 10·1, 8·2 and 9·1, respectively, with murexide as indicator. The error is $<$ 0·5%. When Ni is titrated at pH 9·1, Ca at $<$ 3 times the molar concn. of Ni, and Mg at $<$ 10 times this concn. do not affect the determination; Cd is titrated with **I** at pH 10·2 with catechol violet as indicator. In each case the colour change is very sharp.

K. Saito

2756. **Coulometric determination of hydrogen by oxidation in an internal-electrolysis cell.** C. Schejtanow and J. Kis (Chem.-tech. Inst., Sofia, Bulgaria). *Compt. Rend. Acad. Bulg. Sci.*, 1960, **13** (6), 693–696 (in German).—Electrolytically generated H is rapidly and accurately determined by passing it through the anolyte of an internal-electrolysis cell at const. temp., which oxidises H to H^+. The current passing is proportional to the H content. The solubility of H in the electrolyte affects the results and stirring increases the current.

J. P. Stern

2757. **Deuterium analysis by infra-red spectrophotometry.** R. N. Jones and M. A. Mackenzie (Div. of Pure Chem., Nat. Res. Council of Canada, Ottawa). *Talanta*, 1960, **7** (1–2), 124–126 (in English).—A description is given of modifications in procedure which permit an earlier method (*Ibid.*, 1960, **3**, 356) to be carried out with a Perkin-Elmer spectrophotometer model 137 instead of model 21. Improved procedures for handling volatile and hygroscopic liquids are described.

T. R. Andrew

2758. **Sampling for mass-spectrometric deuterium analysis.** G. Hübner (Inst. f. Physikalische Stofftrennung, Leipzig, Germany). *Kernenergie*, 1960, **3**, 888–890 (in German).—A method is given for measuring ^2H concn. in samples of water of only 3 to 5 mg. The possible errors are discussed. The error caused by the contamination of the samples with atmospheric moisture is highly important. Conditions are described that eliminate these errors.

Nucl. Sci. Abstr.

2759. **Densimetric determination of deuterium and oxygen-18 concentration in water by the falling-drop method.** E. Krell (Inst. f. Physikalische Stofftrennung, Leipzig, Germany). *Kernenergie*, 1960, **3**, 897–902 (in German).—The isotopic analysis of particular elements can be carried out by special methods independently of mass spectrometry. For the determination of the concn. of ^2H or ^{18}O, the falling-drop method is one of the most satisfactory. The theoretical basis of the method is discussed, and an apparatus with a new principle of temp. control, with a constancy of $\pm 0{\cdot}001°$, is described. A new medium, isobutyl benzoate, shows a high constancy and can be used for a range of 0 to 4% of ^2H or ^{18}O. In the range of 1 to 4% the method gives an accuracy of ± 2 μg, corresponding to a relative error of $\pm 0{\cdot}18$ to $0{\cdot}04\%$.

NUCL. SCI. ABSTR.

2760. **Analysis of heavy water by densimetric methods.** Tsing-Lien Tschang (Univ. of Peking, China). *Kernenergie*, 1960, **3**, 892–897 (in German).—The applicabilities of (*i*) the falling-drop method, (*ii*) the temperature-gradient method, and (*iii*) the density-gradient method for the analysis of ^2H_2O are described. For (*i*) a new model of micropipette and new media for the determination of concn. in the range 0 to 100% of ^2H_2O are proposed. For (*ii*) different models of floats were studied and a method for the normalisation of the ^{18}O content by CO_2 for the simultaneous determination of ^2H and ^{18}O was developed. For (*iii*) a new xylene - bromobenzene system for the density-gradient tube is suggested. NUCL. SCI. ABSTR.

2761. **Identification of alkali metals by radial electrochromatographic separation.** C. Bergamini and G. Rapi (Florence Univ., Italy). *Ann. Chim., Roma*, 1960, **50** (10), 1314–1320.—The separations of Li - Na - K and K - Rb - Cs are effected by radial paper chromatography followed directly by electrophoresis, the direction of electrophoretic migration in the former separation being opposite to, and, in the latter separation the same as, that of the initial migration. A buffer soln. of NH_4Cl - HCl diluted with propane-1,2-diol and isopropyl alcohol is used. The Li and Na are rendered visible with zinc uranyl acetate, and the K, Rb and Cs with the sodium salt of dipicrylamine (giving a red coloration with K, violet with Rb and orange with Cs). L. A. O'NEILL

2762. **Method of simultaneous determination of small amounts of sodium and potassium by neutron activation.** L. Adamski, J. Bouzyk and K. Józefowicz (Inst. of Nucl. Res., Polish Acad. Sci., Warsaw). *Nukleonika*, 1960, **5**, 317–327 (in Polish).—The content of these elements was determined in a hydrochloric acid sample obtained from KCl by ion exchange. γ-Spectroscopy and the β-ray absorption method were used. The extrapolated sensitivities were 10^{-7} g of Na and 10^{-5} g of K. The mean error was $<20\%$. NUCL. SCI. ABSTR.

2763. **Metal complexes of β-dioximes. II. Photometric determination of copper employing pentane-2,4-dione [acetylacetone] dioxime.** A. H. I. Ben-Bassat and Y. Sa'at (Dept. of Inorg. and Anal. Chem., Hebrew Univ., Jerusalem, Israel). *Chemist Analyst*, 1960, **49** (4), 108–109 (in English).—The preparation and use of the reagent are described (*cf.* Ben-Bassat *et al.*, *Bull. Soc. Chim. France*, 1960, 948). *Procedure*—To the sample soln. (1 vol. containing 0·5 to 1 mg of Cu per ml) add Na acetate - acetic acid buffer (pH 5·5) (1 vol.), 0·1M-acetylacetone dioxime (2 vol.) and water (1 vol.). Mix, and measure the extinction at 600 mμ, or at 550 mμ if Co and Ni are present; Co can be tolerated up to 9 times, and Ni up to 3 times, the Cu concn. The Cr^{III} concn. in the final soln. must not exceed 0·01M. There is no interference from Mg, Ca, Ba, Ce^{III}, Th, Mn^{II}, Zn, Cd, Al or Zr^{IV}.

W. T. CARTER

2764. **Potentiometric titration of copper with sodium diethyldithiocarbamate.** A. Hulanicki (Dept. of Inorg. Chem., Univ., Warsaw, Poland). *Chem. Anal., Warsaw*, 1960, **5** (6), 881–887 (in English).—*Procedure*—To the test soln. (containing 1·2 to 6·0 mg of Cu) add 20% ammonium sulphosalicylate soln. (5 ml) and 2M-aq. NH_3 - NH_4Cl buffer soln. (pH 9) (10 ml). Dilute to 100 ml and titrate with 0·01M-Na diethyldithiocarbamate, with a silver electrode *vs.* the S.C.E. The end-point is at $\simeq -0{\cdot}15$ V. The silver electrode should be soaked in diethyldithiocarbamate soln. before beginning the titration and immersed for 1 to 2 min. in 5% KCN soln. after each determination. It is recommended that a standard soln. of Cu should be measured before the analysis of samples. The error in the presence of Fe^{3+} (100-fold excess) is $\gtrsim 6\%$. Aluminium does not interfere, but Cd, Zn, Pb, Ni, Co, Ag, Hg^{2+} and Mn do. B. K.

2765. **Use of hydrogen peroxide to effect rapid solution of copper-base alloys in sulphuric acid.** J. Brinn (Northwestern Metal Co., Lincoln, Nebr., U.S.A.). *Chemist Analyst*, 1960, **49** (4), 117 (in English).—Rapid dissolution of copper and copper alloys is possible in a mixture of 30% H_2O_2, conc. H_2SO_4 and water (3:2:4). A cold-water bath is used to moderate the reaction and, when dissolution is complete, H_2O_2 is destroyed by boiling.

W. T. CARTER

2766. **Amperometric determination of metals with thioacetamide. I. Determination of silver.** M. Pryszczewska (Dept. of Gen. Chem., Polytechnic, Szczecin, Poland). *Chem. Anal., Warsaw*, 1960, **5** (6), 931–934.—Aq. thioacetamide soln. (0·0245M) was used for amperometric determination of Ag^+ in aq. $AgNO_3$ soln. (concn. 10^{-4} to 10^{-2}M). The soln. was buffered to pH 9·3. The potential used was $-0{\cdot}4$ V *vs.* the S.C.E., and the dropping-mercury electrode was used as the indicator. The error of the method is $\gtrsim \pm 1{\cdot}4\%$. B. K.

2767. **Chemical determination of trace elements in biological materials. XXII. Co-precipitation of silver with tellurium as carrier.** Kiyoshi Kudo (Chem. Dept., Fac. of Sci., Tohoku Univ., Katahira-cho, Sendai). *J. Chem. Soc. Japan, Pure Chem. Sect.*, 1960, **81** (4), 570–572.—The co-pptn. of Ag (tracer ^{110}Ag) with Te (1 mg) and $SnCl_2$ (3 g) is quant. in boiling N-HCl, but the amount decreases with either increase or decrease in HCl concn., or with the use of mineral acids other than HCl. Co-pptn. does not occur when SO_2 or hydrazine is used as reducing agent. K. SAITO

2768. **Trialkyl thiophosphates. Selective extractants for silver and mercury.** T. H. Handley and J. A. Dean (Anal. Chem. Div., Oak Ridge Nat. Lab., Tenn.). *Anal. Chem.*, 1960, **32** (13), 1878–1883.—Tri-iso-octyl and tri-n-butyl phosphorothioates are highly selective extractants for Ag^+ and Hg^{2+} in HNO_3 medium. From aq. soln. 6M in HNO_3 and

a 0·67M soln. of reagent in CCl_4, the partition coeff. at room temp. are >100 for Ag and 90 for Hg.
D. C. ARMSWORTH

2769. **Decomposition of ferrocyanide in analysis. IV. New spot test for gold.** I. Kraljić (Zagreb Univ., Yugoslavia). *Anal. Chim. Acta*, 1960, **23** (6), 514–517 (in English).—Nitrosobenzene (**I**) and $K_4Fe(CN)_6$ react in the presence of Au^{3+} to produce the violet complex $[Fe(CN)_5.NO.C_6H_5]^{3-}$. *Procedure*—Mix on a spot-plate 1 drop of $K_4Fe(CN)_6.3H_2O$ soln. (0·5%) with 1 drop of a mixture of 4 parts of satd. aq. **I** soln. and 1 part of M-acetate buffer (pH 5·0), and add 1 drop of the test soln. If ≤0·05 μg of Au is present a pink or violet colour is obtained after a short period, or on heating to ≃35°. Interference from Ag and Hg can be eliminated by adding KI. Most common heavy-metal ions, Cl^-, Br^-, SCN^-, oxalate, PO_4^{3-}, $S_2O_3^{2-}$, SO_3^{2-} and CN^- reduce the sensitivity. The test must be carried out in diffused daylight or artificial light.
H. N. S.

2770. **Determination of traces of gold in ores and minerals by spectrographic analysis. II.** F. Macher (Elzett Ironworks and Tech. Univ., Sopron, Hungary). *Magyar Kém. Foly.*, 1960, **66** (12), 507–508.—In the method described for the determination of $1·5 \times 10^{-9}$% of Au in biotite, the Au in a 100-g sample is concentrated into a few mg of lead by chemical means, and the resulting regulus is examined spectrographically. A medium quartz spectrograph is used in conjunction with graphite electrodes and an interrupted d.c. arc. Lead of known Au content serves as a comparison standard, and Cu as internal standard. The intensities of the Au 2676-A and Cu 2724-A lines are used to construct the calibration curve.
B. VASZKO

2771. **Spectrographic analysis of beryllium metal and compounds using barium hydroxide - graphite mixtures.** M. A. Lund and D. L. G. Smith (Chem. Div., A.E.R.E., Harwell, Berks.). AERE-AM 73, 1961. 17 pp.—Procedures are described for the conversion of the sample into BeO, its mixture with a diluent of $Ba(OH)_2$, SnO_2 and graphite (in the proportions BeO 56·6%, graphite 33·3%, $Ba(OH)_2.8H_2O$ 10%, and SnO_2 0·1%), loading into graphite cups and determination of impurities by an arc discharge with a large quartz spectrograph. The method is suitable for the determination of 23 impurities in concn. up to 1000 p.p.m.
T. R. ANDREW

2772. **Direct determination of calcium and magnesium in the presence of iron, aluminium and titanium with ethylenediaminetetra-acetic acid (disodium salt).** Z. Zalessky, J. Debras-Guédon and I. A. Voinovitch (Centre Nat. d'Ét. et de Rech. Céramiques, Paris, France). *Anal. Chim. Acta*, 1960, **23** (6), 523–530 (in French).—In the titration of Ca with EDTA at pH 12, Fe^{3+}, Al^{3+} and Ti^{4+} are masked with tartrate, and calcein is used as indicator. When Ca plus Mg is titrated in ammoniacal soln., with Eriochrome black T as indicator, Fe^{3+}, Al^{3+} and Ti^{4+} are masked with tartrate and triethanolamine. The methods are applied to such materials as natural silicates and cement, after heating the sample with conc. H_2SO_4 and HF until it is soluble in dil. HCl. *Titration of Ca*—Dilute an aliquot of the soln. in dil. HCl that contains ≤0·3 mg of CaO to 50 ml. Add 10 ml of tartaric acid soln. (2%), 10 ml of 4N-KOH and ≃0·1 g of a mixture of calcein (0·1 g) and NaCl (100 g) and titrate with 0·01M- or 0·05M-EDTA (disodium salt) to a pink end-point. *Titration of Ca plus Mg*—To 50 ml of soln., containing ≤1 mg of MgO, add 10 ml of tartaric acid soln. (2%), 10 to 15 ml of triethanolamine, 10 ml of aq. NH_3 (sp. gr. 0·92) and 1 g of a mixture of Eriochrome black T (0·3 g) and NH_4Cl (100 g). Titrate with 0·05M-EDTA (disodium salt) to a blue - violet end-point.
H. N. S.

2773. **Chelatometric determination of calcium and magnesium in minerals.** R. A. Burg and H. F. Conaghan (Chem. Lab., Dept. of Mines, Sydney, N.S.W., Australia). *Chemist Analyst*, 1960, **49** (4), 100–102 (in English).—An improved titration of Ca in the presence of Mg is possible if 1,2-di-(2-aminoethoxy)ethane-*NNN'N'*-tetra-acetic acid (**I**) is used as titrant at pH 13, with Calcon (C.I. Mordant Black 17) as indicator and with tartrate added to prevent the pptn. of Mg. *Procedure*—To the sample soln. (50 ml) add a small amount of ascorbic acid, 10% KCN soln. (2 drops), 10% Na K tartrate soln. (2 ml) and 2M-NaOH (2·5 ml plus 9·5 ml per g of NH_4Cl present). Warm to 40°, add Calcon and titrate with 0·01M-**I**. If the Mg to Ca molar ratio is <1:1, add (before the addition of tartrate) a vol. of 0·1M-$MgCl_2$ equivalent to the total Ca plus Mg present. The total Ca plus Mg is determined by titration with EDTA at pH 10·5 of a separate aliquot, with Eriochrome black T as indicator. The method is applicable to water and to the analysis of rocks and minerals after the removal of the R_2O_3 ppt. Triplicate determinations on a series of samples containing from 0·2 to 98% of $CaCO_3$ and 99·0 to 0·5% of $MgCO_3$ gave mean deviations of 0·2%.
W. T. CARTER

2774. **Determination of calcium and magnesium in high-manganese welding fluxes.** P. Provondra and R. Přibil (Lab. of Anal. Chem., Chem. Inst., Czech. Acad. Sci., Prague). *Chemist Analyst*, 1960, **49** (4), 109–110 (in English).—Manganese can be rapidly separated from Ca and Mg by passing a soln. containing 1,2-diaminocyclohexanetetra-acetic acid (**I**) at pH 4·0 to 4·4 through a column of cationic resin. The complex of Mn with **I** passes through the resin, while the Ca and Mg, which do not form complexes at pH 4·0, are retained and can be eluted with EDTA at pH 12·0. *Procedure*—To the sample soln. containing <150 mg of Mn add a 20% excess of **I** and 0·35 g of NaOH per g of **I**. Adjust to pH 4·0 to 4·4 with Na acetate - acetic acid buffer and dilute to 180 ml with water. Pass the soln. through a column (25 cm × 1 cm) of cation-exchange resin previously treated with Na acetate - acetic acid buffer and wash the column with buffer soln. (3 × 5 ml) and water (50 ml). Elute with 0·05M-EDTA buffered to pH 12·0 (50 ml) and determine Ca and Mg in the eluate by appropriate back-titration procedures. More accurate results for Ca can be obtained by pptn. as oxalate at pH 4·0 to 4·4, destruction of the oxalate with HNO_3 and $HClO_4$ and titration with 0·05M-EDTA, with calcein as indicator.
W. T. CARTER

2775. **Spectrophotometric determination of microgram amounts of calcium.** J. R. W. Kerr (D.S.I.R., Lab. of the Gov. Chemist, Dudley House, Endell St., London, England). *Analyst*, 1960, **85**, 867–870.—The sample soln. (10 ml containing ≥40 μg of Ca) is treated with 1 ml of a NaOH - $Na_2B_4O_7$ buffer soln. (pH 12·6), 0·5 ml of a glyoxal bis-(2-hydroxyanil) reagent soln. and 10 ml of a mixture of ethanol and n-butanol (1:1) in that order. After

30 min. the extinction is measured at 520 mμ, or with an absorptiometer with Ilford No. 604 filters, against water. The calibration soln. of $CaCO_3$ in dil. HCl is adjusted so that 1 ml ≡ 4 μg of Ca. Magnesium and Al do not interfere up to 50 p.p.m., Ba and Sr interfere at 4 p.p.m., and Fe^{3+} up to 50 p.p.m. do not interfere if sufficient KCN is present to prevent pptn. of $Fe(OH)_3$. In concn. up to 500 p.p.m., SO_4^{2-}, Cl^- and NO_3^- have no effect. Results from rain water and sea water are quoted and compared with results determined by titration against EDTA. A. O. JONES

2776. **Rapid determination of alkaline-earth elements. I. Separation of strontium from calcium with *NN*-di(hydroxyethyl)glycine as masking agent.** Susumu Takamoto (Fac. of Sci., Gakushu in Univ., Toshima-ku, Tokyo). *J. Chem. Soc. Japan, Pure Chem. Sect.*, 1960, **81** (6), 915–918.—The difference in stability of the complexes of *NN*-di(hydroxyethyl)glycine (**I**) with Sr and with Ca is so marked that only Sr (0·02 to 0·2 millimole) is pptd. with $(NH_4)_2SO_4$ in the presence of Ca (≃0·2 millimole) and **I** (M, 1·5 ml per 5 ml of the aq. soln.). After heating the mixture for 5 min. in a boiling-water bath, ethanol (3·5 ml for 5 ml) is added to decrease the solubility of $SrSO_4$. The ppt. is separated by centrifuging, washed with aq. ethanol (20%, 2 ml; 40%, 2 × 2 ml), and dissolved in 0·12M-EDTA (2 ml) and conc. aq. NH_3 soln. (0·5 ml), and the soln. is titrated with 0·025M-$Mg(NO_3)_2$ in the presence of Eriochrome black T and an ammonium buffer (pH 10) containing 7% of NH_4Cl (0·5 ml). There is no interference from K, Mg (<0·2 millimole), Na (<1 millimole), NH_4^+ (<3 millimoles), NO_3^-, ClO_4^-, Cl^-, Br^-, I^-, SCN^- or acetate (<1 millimole each). K. SAITO

2777. **Separation of radio-isotopes with ion-exchange membrane. I. Concentration of carrier-free yttrium-90 from strontium-90.** Hidehiro Gotô, Shin Suzuki and Makoto Saito (Inst. Iron, Steel and other Metals, Tohoku Univ., Katahira-cho, Sendai). *J. Chem. Soc. Japan, Pure Chem. Sect.*, 1960, **81** (4), 578–582.—Strontium does not pass through the anion-exchange resin membrane Amberplex A-1 in <0·001M-HCl or an NH_4Cl buffer of pH 3·0, whereas ^{90}Y does, and a separation can thus be effected. The rate of migration of Y increases with increase in electrolytic current across the membrane, which increases with increase in concn. of the electrolyte. K. SAITO

2778. **Determination of low concentrations of radium in water by scintillation counting of electro-deposited decay products of radon.** J. Klumpar, M. Majerová and P. Jirousek (Dosimetric Dept., Inst. Nucl. Res., Acad. Sci., Prague). *Jaderná Energie*, 1960, **6** (12), 404–408.—The apparatus described enables 6·3 × 10^{-14} g of Ra per litre of H_2O to be determined after a 4-day accumulation without preliminary separation. J. ZÝKA

2779. **Use of unithiol for masking zinc, cadmium, mercury, lead and tin in the complexometric determination of strontium and barium.** L. A. Vol'f (S. M. Kirov Leningrad Textile Inst.). *Zavod. Lab.*, 1960, **26** (12), 1353–1354.—Complexes formed by Zn, Cd, Hg, Pb and Sn with unithiol (sodium 2,3-dimercaptopropanesulphonate) (**I**) are more stable than those formed with EDTA. Satisfactory titrations of 10 ml of 0·01N-Sr^{2+} or Ba^{2+} with 0·01N-EDTA (disodium salt) are possible in the presence of 10 ml of a 0·01N soln. of any one of these metals if 10 ml of 0·5% **I** soln., 10 ml of 25% aq. NH_3 and an indicator prepared by mixing 0·1 g of *o*-cresolphthalein complexan, 0·05 g of diamine green B, 0·005 g of methyl red and 1·5 g of NaCl are added, and the soln. is diluted to 100 ml. G. S. SMITH

2780. **Determination of zinc in phosphating [rust-proofing] solutions.** A. T. Kabakov. *Zavod. Lab.*, 1960, **26** (12), 1356.—The soln. (5 ml) containing Zn (2 to 5 g per litre), Fe (8 to 12 g per litre) and PO_4^{3-} (6 to 9 g per litre) is treated with 1 or 2 drops of 25% aq. NH_3 and the ppt. is dissolved by the addition of 2N-HCl. The soln. is diluted to 20 ml with 2N-HCl and passed through a column of ÉDÉ-10p that has been treated successively with 20% NaOH soln., water, and 2N-HCl. The adsorbed Zn is eluted with water and titrated with EDTA (disodium salt). G. S. SMITH

2781. **Gravimetric determination of cadmium with the sodium salt of 1-phenyltetrazoline-5-thione.** C. E. Moore and T. A. Robinson (Loyola Univ., Chicago, Ill., U.S.A.). *Anal. Chim. Acta*, 1960, **23** (6), 533–537 (in English).—1-Phenyltetrazoline-5-thione (**I**) yields with Cd^{2+} a white ppt. that contains **I** and Cd in the ratio 2:1 (24·08% of Cd). The soln. (50 ml) of Cd^{2+} (1 to 40 mg) is heated nearly to boiling and a satd. aq. soln. of **I** is added dropwise, with stirring. The ppt. is filtered off on a sintered glass crucible, washed with water, dried at 110°, and weighed. Recorded errors range from 0 to 0·3 mg of Cd. Cadmium can be determined in the presence of Mn and Mg, but Zn, Pb, Co, Ni, Ag, Hg, Cr, Cu, Sn, Sb and Bi yield ppt. H. N. S.

2782. **Method for the detection of mercury by radioactivation analysis. [Radioactivation Analysis Symposium. Vienna, June 1959.]** K. Ljunggren and T. Westermark (Royal Inst. of Technol., Stockholm 70, Sweden). *Pure and Appl. Chem.*, 1960, **1** (1), 127–133 (in English).—The method has been applied to biological materials, aluminium, cellulose and grain. To avoid loss of Hg by evaporation in the reactor, the samples and standards are sealed in quartz ampoules; the 77-keV γ-radiation from ^{197}Hg is measured after a cooling period of two days. For an irradiation of 3 days at 1 to 2 × 10^{12} neutrons per sq. cm per sec., the sensitivity limit is 0·03 μg. G. J. HUNTER

2783. **Simplified colorimetric determination of mercury.** S. S. Yamamura (Atomic Energy Div., Phillips Petroleum Co., Idaho Falls). *Anal. Chem.*, 1960, **32** (13), 1896–1897.—A modified Friedeburg method (*Anal. Abstr.*, 1955, **2**, 2046) is described. The sample (50 to 300 μg of Hg) is boiled with conc. HNO_3 to oxidise Hg^I, and after cooling and diluting the soln. to 50 ml an aliquot (≯10 ml) is treated with a specified EDTA - chloroacetic acid - ammonium citrate soln. (pH 3·8 to 4·0) and the Hg is extracted with a 0·0011% soln. of dithizone in xylene. The Hg is back-extracted, first into 6M- and then into 3M-HCl, the aq. phase is treated with a specified EDTA - chloroacetic acid - aq. NH_3 soln. (pH 7·0) and the Hg is extracted with a 0·0011% soln. of dithizone in CCl_4. The extinction of the CCl_4 phase is measured at 510 mμ. R. E. E.

2784. **Spectrographic determination of boron.** M. Kulcsár and I. Benkő (Res. Inst. for Heavy Chem. Ind., Veszprém, Hungary). *Magyar Kém. Foly.*, 1960, **66** (12), 508–510.—The method proposed

for determining 0·001 to 0·1 p.p.m. of B in high-purity silicon is based on the concentration of the B of a 20-g sample into 20 mg of carrier material by chemical means, followed by examination of the concentrate by the ISP-22 spectrograph. The following compounds were investigated as possible carrier materials, both singly and in combination—SiO_2, NaCl, NH_4Cl, ZnO, $BaCl_2$, $CaCO_3$ and Al_2O_3; known amounts of B were added as H_3BO_3. The effects of current density and electrode polarity were studied. It is claimed that 0·001% of B in the concentrate can be determined by the use of Sb as internal standard, and SiO_2 as carrier material, in conjunction with copper electrodes and an interrupted a.c. arc, and 0·0001% of B can be determined by the use of Zr as internal standard and NH_4Cl - ZnO (1 : 1) as carrier material, with B-free graphite electrodes and a d.c. arc. In each case the error is within ±10%. B. VASZKO

2785. **Statistical analysis of spectrophotometric determinations of boron.** F. W. Lima, C. Pagano and B. Schneiderman [Inst. de Energia Atomica, Caixa Postal 11049 (Pinheiros), São Paulo, Brazil]. *Analyst*, 1960, **85**, 909–915.—Different amounts of H_3BO_3 soln. (10 μg of B per ml) were placed in quartz crucibles of different vol. with 0·1 ml of 0·1N-NaOH if the effect of alkalinity was being studied. The liquids were evaporated, 1 ml each of trichloroacetic acid soln. (163 g per litre) and ethanolic curcumin soln. (0·125% w/v) were added and the crucibles were dried to constant wt., either at 55° or at 100°. The residues were dissolved in ethanol and the extinctions were measured at 550 mμ. A 2^3 factorial design was used with three factors, *viz*, vol. of crucible (*v*), amount of H_3BO_3 and temp. From the results it is concluded that (*a*) extinctions measured for nominally identical samples differ significantly on different days; (*b*) the value of *v* affects the results unless alkali is added before evaporation, but this effect is independent of the amount of B present and thus only one size of crucible should be used; (*c*) the temp. of drying does not affect the results; (*d*) the relative error for each reading is ≃3·4 for the range 2·5 to 7·5 μg of B (alkali not added) and 2·2 for the range 0·33 to 1 μg of B (alkali added); (*e*) Beer's law is obeyed for amounts of B up to 7·5 μg per crucible. A. O. JONES

2786. **Determination of boron in silicon by isotope dilution.** D. C. Newton, J. Sanders and A. C. Tyrrell (The Plessy Co. Ltd., Caswell Res. Lab., Towcester, Northants, England). *Analyst*, 1960, **85**, 870–877.—To the silicon sample, cleaned and etched by a specified procedure, are added 20 ml of pre-electrolysed (*i.e.*, B-free) NaOH soln. and 1 μg of ^{10}B tracer, and the mixture is heated for 30 min. on a steam bath. The cooled soln. is transferred by means of a polyethylene pipette and 10 ml of water to the anode compartment of an electrolytic cell containing a cation-exchange membrane to remove Na^+ during electrolysis. Sodium hydroxide soln. (1% w/v) is placed in the cathode compartment. Electrolysis is started at 0·7 amp. and ≃5 V and continued for ≃2·25 hr., the voltage being increased as the current falls until the final conditions are 25 mA at 50 V. The anolyte is evaporated to dryness, the residue is crushed, re-dissolved in 2 ml of water and again evaporated. The final residue is extracted with the minimum amount of water (≃0·01 ml), and the B content is determined in a mass spectrograph. A. O. JONES

2787. **Spectrophotometric determination of boron in molybdenum alloys with carmine.** D. G. Higgs (The War Office, Armament Res. and Development Estab., Fort Halstead, Kent, England). *Analyst*, 1960, **85**, 897–904.—A mixture of 0·2 g of sample, 10 ml of water, 5 ml of HCl and 12 to 15 drops of HNO_3 is heated gently under reflux until dissolution is complete, drops of HNO_3 being added if necessary. After the addition of 0·5 g of urea, the soln. and washings of the condenser are diluted to 100 ml. An aliquot (5 ml) in a porcelain crucible is made alkaline to phenolphthalein with Na_2CO_3 and evaporated to dryness. The residue is dissolved in water (2 ml), 10 ml of conc. H_2SO_4 is added, the cooled liquid is transferred to a 25-ml flask and the crucible is rinsed with 10 ml of carmine soln. (0·05% w/v in H_2SO_4) and then with H_2SO_4. The liquid is adjusted to vol. with H_2SO_4 and after 90 min. the extinction is measured at 610 mμ against a blank soln. prepared from 0·2 g of B-free molybdenum and referred to a calibration graph. The mean error over the range 0·01 to 0·2% is ±0·006% of B. A. O. JONES

2788. **Rapid method for dissolving highly pure aluminium for analytical purposes.** P. M. Houpt and R. A. F. Winia (Hunter Douglas Holland Lab., 2 Piekstraat, Rotterdam, The Netherlands). *Analyst*, 1960, **85**, 924.—Aluminium (99·9% purity) can be rapidly dissolved without heating in a soln. consisting of 10 ml each of 36% HCl and 30% H_2O_2 and 1 to 5 drops of 10% (w/v) $CuCl_2$ soln. If the addition of Cu is undesirable, dissolution of the metal in HCl alone is very rapid when the material is in contact with an electropositive element (*e.g.*, platinum or gold). When a platinum crucible is used as a container, the aluminium dissolves rapidly in 10 ml of conc. HCl if a few drops of H_2O_2 are added as a depolariser. A. O. JONES

2789. **Analytical application of embelin. II. Determination of aluminium and beryllium and their separation.** C. Bheemasankara Rao and V. Venkateswarlu (Andhra Univ., Waltair, India). *Z. anal. Chem.*, 1961, **178** (4), 277–280 (in English).—With embelin (2,5-dihydroxy-3-undecyl-1,4-benzoquinone) (**I**), aluminium soln. at pH 4 to 4·5 give a bluish-violet complex that is determined either by direct weighing or as Al_2O_3 after ignition, while Be at pH 6·5 to 7 gives a pale-pink complex soluble in ethanol. *Procedure for the separation of Al from Be*—To a soln. of the chlorides of Al and Be is added 2 g of NH_4Cl, the soln. is diluted to 100 ml, adjusted to pH 4·0 to 4·5 and warmed to ≃60°; 1% ethanolic **I** soln. is added dropwise (12 to 15 ml per 0·01 g of Al_2O_3), with stirring. The mixture is digested on a steam bath for ≃30 min. The ppt. is then allowed to settle, and is either collected on Whatman No. 41 paper, washed with cold water and then ignited in a platinum crucible and weighed as Al_2O_3, or is collected in a sintered glass crucible, washed with 2% ethanolic embelin soln., then with cold water and finally with ethanol. It is then dried at 105° to 110° and weighed. To determine Be in the filtrate, 15 ml of M-ammonium acetate is added and the pH is adjusted to 6·5 to 7·0 with dil. aq. NH_3. The soln. is warmed to 60° and **I** is added as described above (13 to 15 ml per 0·01 g of BeO). The mixture is heated on a steam bath until most of the ethanol is removed. The ppt. is then allowed to settle, collected on Whatman No. 42 paper, washed with H_2O containing ammonium acetate, ignited in a platinum crucible and weighed as BeO. Results are given. B. B. BAUMINGER

2790. **Systematic analysis of 'zone-refined' aluminium and iron after irradiation in an atomic pile. [Radioactivation Analysis Symposium. Vienna, June 1959.]** P. Albert (C.N.R.S., 15 rue G. Urbain, Vitry, Seine, France). *Pure and Appl. Chem.*, 1960, **1** (1), 111–119 (in French).—Analyses are described in which samples (1 g) of metal were irradiated for one week in a flux of $\simeq 10^{12}$ neutrons per sq. cm per sec. Group-separation procedures were used and results for 48 elements are tabulated; for about 40 of these the limits of sensitivity are between 0·1 and 0·0001 p.p.m. G. J. HUNTER

2791. **Flame-photometric determination of alkali-metal contaminants in aluminium.** E. Pungor and E. Zapp (L. Eötvös Sci. Univ., Budapest). *Magyar Kém. Foly.*, 1960, **66** (12), 523–525.—Trace amounts (10^{-3} to 10^{-4}%) of Na and K present in aluminium metal were determined with a Beckman DU spectrophotometer and a H - O flame. The samples were dissolved in distilled HCl contained in quartz vessels. The effect of Al on Na, K and Li emissivities was investigated. Calibration curves were prepared by the addition method; other elements present in the metal sample did not interfere.

B. VASZKO

2792. **Determination of indium and tin in glycerol plating baths.** E. P. Cocozza (Philco Corp., Lansdale Div., Lansdale, Pa., U.S.A.). *Chemist Analyst*, 1960, **49** (4), 124–126 (in English).—Indium is determined by the procedure of Flaschka and Abdine (*cf. Anal. Abstr.*, 1957, **4**, 361), in which Sn is masked with NaF and the In is titrated with EDTA at pH 2·5, with Cu - EDTA soln. and 1-(2-pyridylazo)-2-naphthol (PAN) as indicator. Tin is determined iodimetrically on another portion of sample after reduction with iron powder. A sample containing 5·18% of In and 1·24% of Sn gave results for both components with an accuracy better than 1%. W. T. CARTER

2793. **Modified a.c. polarograph with a circuit for charging-current compensation and its application for the determination of traces of cadmium and lead in indium metal.** M. Ishibashi, T. Fujinaga and A. Saito (Chem. Inst., Fac. Sci., Univ., Kyoto, Japan). *Coll. Czech. Chem. Commun.*, 1960, **25** (12), 3387–3390 (in English).—The modified polarograph is described. The reduction of In in $HClO_4$ proceeds irreversibly at the dropping-mercury electrode and therefore causes no rise of current in a.c. polarography; Cd and Pb are reduced reversibly in the same electrolyte and may therefore be determined without preliminary separation from In. *Procedure*—Dissolve the sample (0·5 to 1·0 g) in 30% $HClO_4$ (4 to 8 ml) and conc. HNO_3 (5 drops), evaporate to fumes of $HClO_4$, add 30% $HClO_4$ (1 to 2 ml), evaporate again and dissolve the residue in $HClO_4$. Dilute to 10 ml with M-$HClO_4$. Record the polarogram of an aliquot from − 0·3 to − 0·8 V *vs.* the S.C.E. A. J. BENNETT

2794. **Theoretical considerations on space diagrams of sensitivity for thallium detection in the presence of silver.** J. Ramírez-Muñoz and L. Capacho-Delgado (Inst. of Pure and Appl. Res., Div. of Sci. Res., Ind. Univ. of Santander, Bucaramanga, Colombia). *Microchem. J.*, 1960, **4** (4), 515–526.—The iodide test for Tl, in which KCN and Na K tartrate soln. are used to mask Ag and Pb, is examined. Three-dimensional diagrams show the effects on the sensitivity of the test of variable amounts of masking agents and interfering elements, all in the same soln. The following was found to be the most convenient procedure. To one drop of the neutral or slightly acid test soln. in a micro test-tube add one drop of 20% Na K tartrate soln., one drop of 10% KCN soln. and one drop of 15% KI soln. An insol. yellow ppt. shows the presence of Tl. The sensitivity, pTl (= −log[Tl]) is in the range 4·6 to 4·7. A. J. BENNETT

2795. **Microchemical detection of thallium with the aid of an ion-exchange resin bed.** Kiyoshi Kato and Hidetake Kakihana (Nagoya Municipal Tech. High School, Nakagawa-ku). *J. Chem. Soc. Japan, Pure Chem. Sect.*, 1960, **81** (3), 452–454.—When Dowex 1-X1 (iodide form) is placed in an HNO_3 soln. of pH 1 containing Tl^{I}, a yellow coloration appears after 1·5 hr. The limit of detection is 0·01 μg and the concn. limit 1 in $1·3 \times 10^6$. Copper, Ag, Hg, Ti, Pb, Bi, UO_2^{2+}, VO_3^-, Fe^{III} and $S_2O_3^{2-}$ interfere. The sensitivity of the alizarin method was not increased by the use of an ion-exchange resin. K. SAITO

2796. **Detection of the rare-earth elements by the Zeiss Q24 spectrograph.** I. Kerekes (Telecommun. Ind. Res. Inst., Budapest). *Magyar Kém. Foly.*, 1960, **66** (12), 510–513.—The growing importance of the detection and determination of the rare-earth elements, and the physico-chemical principles and analytical difficulties involved in their spectrographic analysis are discussed. Spectrum charts for the following elements are illustrated—Sc, Y, La, Ce, Pr, Nd, Pm, Sm, Eu, Gd, Tb, Dy, Ho, Er, Tm, Yb and Lu, and the method used is described. The wavelength range is 2600 to 5600 A. The wavelengths of the lines, the interfering elements and the relative intensities of the lines are shown on the charts, which also contain a comparison spectrum of Fe. The limit of detection is $\simeq 0·001$% of each element. B. VASZKO

2797. **Spectrophotometric analysis of rare-earth-metal mixtures.** C. G. Schultz (Michigan Chem. Corp., St. Louis, Mich., U.S.A.). *U.S. Atomic Energy Comm.*, Rep. TID-6423, 1957. Decl. Feb. 1959. 6 pp.—A method of analysis is described by which Pr, Nd, Sm, Dy, Ho, Er, Tm and Yb can be determined. Determination of the remainder of the rare-earth metals by difference is complicated by the fact that more than one of those exhibiting no absorption spectrum may be present. The most satisfactory results are obtained with the use of $HClO_4$ soln. Nitrate soln. give high results in many cases. The presence of Ce affects both the solubility and the measurability of the samples.

NUCL. SCI. ABSTR.

2798. **Formaldoxime as an analytical reagent. II. Colorimetric determination of cerium.** Z. Marczenko and J. Minczewski (Dept. of Anal. Chem., Polytech., Warsaw, Poland). *Chem. Anal., Warsaw*, 1960, **5** (6), 903–916.—The method is based on the reaction of Ce^{3+} with formaldoxime in alkaline soln., by which a brown complex of Ce^{4+} with formaldoxime is formed. *Procedure*—After the usual oxidation and removal of Si, dissolve the residue in 50 ml of $HClO_4$ soln. (1:25). Precipitate hydroxides with aq. NH_3 in the presence of H_2O_2, filter off and dissolve the ppt. in N-HCl (10 ml), add H_2O (25 ml) and satd. oxalic acid soln. (30 ml), heat and set aside for 3 to 4 hr. Collect the ppt., wash it with 1% oxalic acid soln., dissolve it in 2N-HCl (10 ml) and add H_2O to 100 ml. To 10 ml of this soln.

add M-formaldoxime (5 ml), neutralise with N-NaOH, add more N-NaOH (5 ml) and H_2O to 50 ml. After 10 min. measure the extinction at 330 mμ with H_2O as a blank. Compare the result with a calibration curve. EDTA, F^-, PO_4^{3-}, Ni, Mn, Fe, V, Cu and Co interfere. The method was used for the determination of Ce in apatite concentrates.
B. K.

2799. **Analysis of ores for yttrium by X-ray fluorescence.** C. G. Schultz (Michigan Chem. Corp., St. Louis, Mich., U.S.A.). *U.S. Atomic Energy Comm.*, Rep. TID-6432, 1957. Decl. Feb. 1959. 9 pp.—In order to obtain a rapid indication of the presence of yttria earths in ores, a method was devised by which the yttrium content of the ores is determined by X-ray fluorescence, with Sr as internal standard. Analyses of ores of known yttrium content were made, and recovery and dilution experiments run, with average deviations ranging from ±2% at the 35% level to ±0·1% at the 5% level. Thallium, Sb and Sn may interfere to a slight extent if present in high concn.; Pb, Rb and iodine will interfere at low concn. All these will tend to give a high yttrium value. Niobium exerts a significant effect even at low concn., resulting in a low yttrium value. If these elements are present, it is necessary to use a spectrographic technique.
NUCL. SCI. ABSTR.

2800. **Spectrographic analysis of impurities in pure yttrium oxide (revised method).** A. B. Whitehead (Michigan Chem. Corp., St. Louis, Mich., U.S.A.). *U.S. Atomic Energy Comm.*, Rep. TID-6421, 1958. Decl. Feb. 1959. 16 pp.—A spectrographic method for the determination of Dy_2O_3, Tb_4O_7 and Gd_2O_3 in high-purity yttrium oxide and the quantitative determination of 16 other impurities was devised. The region 2550 to 4350 A is recorded in the second order. Samples are mixed with graphite powder (1:1) and burned in a graphite electrode.
NUCL. SCI. ABSTR.

2801. **Reduction of europium and terbium at a dropping-mercury electrode.** Akio Iwase (Fac. of Arts and Sci., Yamagata Univ., Koshirakawa-cho). *J. Chem. Soc. Japan, Pure Chem. Sect.*, 1960, **81** (5), 739–744.—Holleck's findings on the polarogram of Eu in 0·1M-LiCl at pH 2 to 5 (*Z. Naturf.*, 1947, **26**, 81) were re-examined. The first wave at −0·67 V *vs.* the S.C.E. (at pH 4·0) shifts towards a more negative region with decrease in pH in the presence of gelatin; in the absence of gelatin the $E_{\frac{1}{4}}$ value is independent of pH (−0·625 V). Both the first and the second wave are diffusion-controlled and the limiting current is proportional to concn. both in the presence and absence of gelatin. In the same basal soln., Tb gives only one diffusion-controlled wave at −1·818 V, irrespective of pH, and the wave height is proportional to concn. in the presence of gelatin.
K. SAITO

2802. **Determination of microgram amounts of cyanide by means of impregnated filter-paper.** W. Kiełczewski and J. Tomkowiak (Dept. of Gen. Chem., School of Agric., Poznań, Poland). *Chem. Anal., Warsaw*, 1960, **5** (6), 889–892.—*Procedure*—Place spots of test soln. on Whatman No. 1 or S. & S. No. 2043b paper impregnated with Ag dithizonate and develop the chromatogram with H_2O - 0·1N-KOH (40:1). The area of the white spot (on a violet - pink background) is proportional to CN^- concn. Refer the result to a calibration curve. Details of the impregnation of the paper and the procedure are given; SCN^-, Cl^- and Br^- do not interfere.
B. K.

2803. **Spectrographic analysis of silicon.** L. Vecsernyés (Res. Inst. for Telecommun., Budapest). *Magyar Kém. Foly.*, 1960, **66** (12), 513–516.—A method for the analysis of semi-conductor silicon is described in which the following 18 elements were determined simultaneously without previous concentration—Al, As, Au, B, Ba, Ca, Co, Cu, Fe, In, Mg, Ni, Pb, Sb, Sn, Ti, W and Zn. The range of concn. investigated was in general 5×10^{-7} to 5×10^{-3} g per g of silicon. A medium quartz spectrograph was employed, an interrupted a.c. arc being maintained across graphite electrodes in a protective atmosphere of argon. Silicon was used as internal standard. Line pairs and the analytical limits of the method are indicated. Two methods for the preparation of comparison standards are given.
B. VASZKO

2804. **Volumetric determination of large amounts of silicon in alloys.** F. Wagner (Süddeutsche Kalkstickstoff-Werke A.-G., Trostberg, Werk Hart/Alz.). *Z. anal. Chem.*, 1960, **178** (1), 34–38 (in German).—Velken's method (*Anal. Abstr.*, 1958, **5**, 4033) is used for the volumetric determination of Si in Fe - Si, Ca - Si, Mn - Ca - Si, and Mg - Ca - Si systems; it is based on the formation of K_2SiF_6 and titration with 0·25N-NaOH, with phenolphthalein as indicator. *Procedure for Fe - Si*—To the sample (0·1 to 0·3 g), in a polyethylene beaker, is added conc. HNO_3, the mixture is cooled in ice and conc. HF is added slowly. When dissolution is complete, 20% KCl soln. is added and the Si is pptd. as K_2SiF_6. The ppt. is filtered off, washed with KCl soln. and then titrated with 0·25N-NaOH. *For Ca - Si*—The sample is fused with a mixture of Na_2CO_3 and Na_2O_2 in a nickel crucible. The cold melt is dissolved in a little water and dil. HCl. Then conc. HNO_3 and solid KCl are added, the mixture is cooled, and HF is added dropwise. The ppt. is washed and titrated as described above. It is shown that the accuracy is often higher than that of the standard gravimetric method, and the max. deviation from the mean is ±0·27%.
B. B. BAUMINGER

2805. **Determination of trace-element diffusion in quartz and in germanium. [Radioactivation Analysis Symposium. Vienna, June 1959.]** G. Leliaert (Centre for Nuclear Energy, Mol, Belgium). *Pure and Appl. Chem.*, 1960, **1** (1), 121–126 (in English).—Two problems are considered. In the determination of the distribution of Hg in quartz tubes the sample is irradiated for one week in a flux of 5×10^{12} neutrons per sq. cm per sec., and, after the inside of the tube has been dissolved, the Hg is chemically separated and the 77-keV γ-radiation from ^{197}Hg is measured and compared with that from a standard. In the determination of the distribution of P and As in germanium, the sample (4 g) is irradiated for 24 hr. in a neutron flux of 10^{13} per sq. cm per sec. and, after the sample has been dissolved, the P and As are separated, and the activities due to ^{76}As and ^{32}P are measured.
G. J. HUNTER

2806. **Volumetric determination of germanium.** G. J. Abel, jun. (American Smelting and Refining Co., S. Plainfield, N.J.). *Anal. Chem.*, 1960, **32** (13), 1886–1887.—Germanium is separated from interfering elements by distillation from HCl containing $KMnO_4$. The distillate is received in 30% HCl

containing NaH_2PO_2 and HBr. The liquid in the receiver is boiled in a flask equipped with a siphon, and cooled with the end of the siphon immersed in satd. $NaHCO_3$ soln. This reduces the Ge to Ge^{II}, which is then immediately titrated with standard KIO_3 - KI soln., with starch as indicator. The precision and accuracy are good. Directions for the dissolution of various types of sample, and for the standardisation of the titrant, are given.

R. E. E.

2807. **Polarographic study of bivalent germanium.** G. Sauvenier and G. Duyckaerts (Liège Univ., Belgium). *Anal. Chim. Acta*, 1960, **23** (6), 569–574 (in French).—Quadrivalent Ge is reduced to Ge^{II} by heating in aq. H_3PO_2 and the polarography of the Ge^{II} is investigated in a medium containing an excess of the acid. In 1·09N-H_3PO_2, $E_{\frac{1}{2}}$ for the cathodic wave is $-0{\cdot}515 \pm 0{\cdot}002$ V, and that for the anodic wave is $-0{\cdot}149 \pm 0{\cdot}003$ V, *vs.* the S.C.E. Diffusion-current constants are also determined. Further studies, covering the influence of pH, provide information on the formation and constitution of hypophosphite and oxalate complexes of Ge.

H. N. S.

2808. **Separation of germanium and antimony by elution chromatography on a cellulose column.** A. M. Ghe (Bologna Univ., Italy). *Ann. Chim., Roma*, 1960, **50** (10), 1321–1332.—The sample soln. is applied to a cellulose column. Elution with pyridine quantitatively removes the Ge, which is then determined colorimetrically with phenylfluorone (at 510 mμ). The residual pyridine is removed by washing with benzene, and the Sb is eluted with H_2O and determined colorimetrically with phenylfluorone (at 530 mμ). The separation is effective for Ge to Sb ratios of 1:1 to 100:1.

L. A. O'Neill

2809. **Complexometric determination of lead in fluoroboric acid electrolyte.** I. M. Yurist and P. G. Shakhova. *Zavod. Lab.*, 1960, **26** (12), 1354–1355.—An aliquot (10 ml) of the filtered electrolyte diluted 1:10 is treated with 5 or 6 drops of 30% H_2O_2, 25 ml of 4% NaF soln. and 50 ml of water, and boiled for 15 min. The cooled soln. is then treated with 10 ml of 0·1N-EDTA (disodium salt), 0·2 g of a mixture of Chromogen black (C.I. Mordant Black 11) and NaCl (1:99) and a buffer soln. (54 g of NH_4Cl and 350 ml of aq. NH_3 per litre) (5 ml more than that sufficient to turn the soln. blue), and the excess of EDTA is determined by titration with 0·05N-$ZnSO_4$; Sn does not interfere.

G. S. Smith

2810. **Cation-exchange studies of lead(II) on Dowex 50W-X8. Separation from mixtures.** S. M. Khopkar and A. K. De (Jadavpur Univ., Calcutta, India). *Talanta*, 1960, **7** (1–2), 7–11 (in English).—The elution of Pb^{II} from Dowex 50W-X8 has been studied with 1M to 6M-HNO_3, 1M to 4M-ammonium acetate, 1M to 2M-$NaNO_3$, 5% tartaric acid, 0·01M-EDTA (disodium salt), and 2M-$NaNO_3$ with 0·5M-HNO_3. Separation from Sr, Ba or Al is achieved by elution of the Pb with M-ammonium acetate, and from Ce^{IV} and Zr by treatment of the soln. with 5% of citric acid at pH 2·7, passage through the resin, and elution of the Pb with M-ammonium acetate. Lead may be separated from Bi, Fe^{III} or Th by treatment of the soln. with 0·01M-EDTA at pH 2 and passage through the resin, which retains the Bi, Fe^{III} and Th, the Pb appearing in the percolate. Recoveries of Pb ranged from 94 to 102%.

T. R. Andrew

2811. **Separation of tin by benzene extraction of stannic iodide.** D. D. Gilbert and E. B. Sandell (Univ. of Minnesota, Minneapolis, U.S.A.). *Microchem. J.*, 1960, **4** (4), 491–500.—For effective extraction, Sn must be present as undissociated SnI_4; this is favoured by high iodide and hydrogen-ion concn. The use of $HClO_4$ and $NaClO_4$ minimises the formation of stannic complexes. Chloride and F^- interfere and must be removed by evaporation with H_2SO_4. *Procedure*—To soln. containing 1 to 50 μg of Sn^{IV} add H_2SO_4 and evaporate to fumes so that the concn. of H_2SO_4 after dilution is $<0{\cdot}5$M. Make the soln. 1·5M in $HClO_4$, 0·5M in NaI and 3M in $NaClO_4$, the total vol. being 15 to 30 ml. Extract with 5 ml of benzene. Wash the benzene extract with 5 ml of an aq. soln. having the same concn. of $HClO_4$, NaI and $NaClO_4$ as the original aq. phase, to reduce the concn. of elements co-extracted with Sn. Shake the benzene extract with 0·25M-H_2SO_4 (5 ml) and heat on a steam bath to remove benzene. If 10 to 50 μg of Sn is present add 30 ml of buffer soln. (5 g of K H phthalate and 17·5 ml of N-HCl per litre), 1% gum acacia soln. (1 ml) and a 0·01% alcoholic soln. of phenylfluorone (10 ml). Dilute to 50 ml and measure the extinction at 510 mμ. If <10 μg of Sn is present add buffer soln. (10 ml), gum acacia soln. (1 ml) and phenylfluorone soln. (5 ml), dilute to 25 ml and measure the extinction at 570 mμ.

A. J. Bennett

2812. **Direct polarographic determination of tin in presence of lead, thallium, arsenic and antimony. Determination of small amounts of tin in lead.** M. Cyrankowska (Central Lab. of Accumulators and Cells, Poznań-Starołęka, Poland). *Chem. Anal., Warsaw*, 1960, **5** (6), 851–861.—*Procedure*—Dissolve the sample (0·1 to 10 g) in conc. H_2SO_4, dilute with H_2O, cool and filter. Evaporate an aliquot of the filtrate nearly to dryness, add 2N-HCl (5 to 10 ml), cool and add 0·1N-$KMnO_4$ to a violet colour. Boil for a few minutes, then cool. Make the soln. 0·01M in EDTA and 2·7M in NaCl and adjust the pH to 5 with NaOH. Acidify the soln. to pH 2 with conc. HCl, add 0·5% theophylline soln., remove oxygen and record the polarogram; $E_{\frac{1}{2}} = -1{\cdot}03$ V *vs.* the S.C.E. Prepare a standard curve by the same procedure. Thallium, Cd, Bi, Cu, As^{5+}, Sb^{5+}, Ni^{2+}, Zn^{2+}, Co^{2+}, Mn^{2+}, Al^{3+} and Cr^{3+} do not interfere; 0·0005% of Sn can be determined in lead; the relative error is $>6\%$.

B. K.

2813. **Chelatometric titration of titanium in the presence of niobium and tantalum.** E. Lassner and R. Scharf (Metallwerk Plansee A.-G., Reutte, Tirol, Austria). *Talanta*, 1960, **7** (1–2), 12–17 (in German).—1,2-Diaminocyclohexanetetra-acetic acid (**I**) forms a complex with peroxytitanyl ion, while no such complex is formed by Nb or Ta. The strongly acid soln. in H_2SO_4 (5 to 20 mg of Ti) is treated with 1 to 2 ml of H_2O_2 (30%) and a measured excess of 0·05M-**I**; 2 to 3 g of Na acetate is added, the soln. is diluted to 150 ml, the pH is adjusted to 5·0 to 5·2 with aq. NH_3 and the soln. is heated to 70° to 90°. To the hot soln. are added 7 or 8 drops of 0·1% ethanolic 1-(2-pyridylazo)-2-naphthol soln. and an excess of 0·05M-$CuSO_4$ (colour change from yellow to red - violet); the soln. is then titrated with 0·05M-**I** to the yellow end-point. Results for 1 to 20 mg of Ti show an average error of 0·025 mg in the presence of up to 100 mg of Ta and 50 mg of Nb.

T. R. Andrew

2814. **Modified tin-flux technique for the determination of oxygen in titanium.** C. Venkateswarlu and M. W. Mallett (Atomic Energy Estab., Trombay,

India). *Anal. Chem.*, 1960, **32** (13), 1888–1890. —A modification of the Walter method (*Brit. Abstr. C*, 1950, 319) is described. Oxygen is extracted at 1950° without using graphite chips. A graphite crucible is packed with 200-mesh graphite powder in a Vycor thimble and 1 g of tin is added.

D. C. ARMSWORTH

2815. **Analysis of pure metals. Determination of vanadium as an impurity in titanium.** M. B. Shustova and V. A. Nazarenko (Inst. of Gen. and Inorg. Chem., Acad. Sci., UkrSSR). *Zavod. Lab.*, 1960, **26** (12), 1339–1341.—The method is based on the catalytic acceleration by V of the oxidation of aniline by $KClO_3$ in the presence of 8-hydroxyquinoline (**I**) as an activator. The interference of Ti is prevented by the use of ammonium tartrate and extraction of the coloured reaction product by ethyl acetate. The sample (0·1 g) is dissolved by treatment with 2 ml of 40% HF and a few drops of conc. HNO_3, followed by addition of 3 ml of conc. H_2SO_4 and evaporation to fuming. The residue is dissolved in water, 10 ml of 10% ammonium tartrate soln. is added, and the soln. is neutralised with NaOH soln. (yellow with methyl orange). After the addition of 2 ml of a 2% soln. of **I** in N-acetic acid, the diluted soln. (100 ml) is extracted with isoamyl alcohol (2 × 10 ml). The combined extracts are washed first with a washing liquid prepared by adding 2 ml of 2% **I** soln. and 10 ml of 10% ammonium tartrate soln. to 200 ml of water, and removing the extractable matter by shaking with 10 ml of isoamyl alcohol. The dissolved matter is then extracted into HNO_3 (1:1) (2 × 2·5 ml with the addition of 0·5 ml of 30% H_2O_2), the soln. is evaporated to dryness, and the residue is ignited at ≯600°, then treated with 2 ml of HF and evaporated to dryness, followed by dissolution in 5 ml of 10% ammonium tartrate soln. The soln. is treated with 2 ml of satd. $Na_4P_2O_7$ soln. and neutralised with dil. HCl in the presence of two drops of 0·01% methyl orange soln. It is then treated with 10 ml of 5% aniline sulphate soln., 5 ml of satd. $KClO_3$ soln. and 2 ml of a 2% soln. of **I** in N-acetic acid, and diluted to 50 ml. The soln. is heated on a boiling-water bath for 30 min., the heating is then discontinued and the soln. is left on the bath for 1 hr. It is then cooled to between 15° and 17° and extracted with 7 ml of ethyl acetate. The extract is mixed with a further 3 ml of ethyl acetate and 0·25 ml of ethanol and the extinction is measured at 390 or 465 mμ. A blank is carried out at the same time. The content of V is found by use of a calibration curve. The minimum content that can be determined with a 0·1-g sample of titanium metal is 5×10^{-5} %. Interference is caused by the presence of Mo. G. S. SMITH

2816. **Determination of titanium tetrachloride in organic solvents.** Toshio Takiguchi, Yasuaki Nakaido and Yoshiharu Kubota (College of Tech., Gumma Univ., Kiriu). *J. Chem. Soc. Japan, Ind. Chem. Sect.*, 1960, **63** (5), 786–787.—Titanium tetrachloride (1 mole) in an organic solvent (amyl acetate, ethyl ether, propanol or hexane) reacts with aniline (4 moles) in ethyl ether to produce aniline hydrochloride in 98% yield; this is dissolved in ethanol and titrated with $AgNO_3$ (0·02 or 0·05N), with dichlorofluorescein as indicator. The error due to hydrolysis of $TiCl_4$ can thus be eliminated. The deviation is <2·4% for 10 to 200 mg of $TiCl_4$.

K. SAITO

2817. **Spectrophotometric determination of titanium in uranyl nitrate by means of phenylfluorone.** I. Pietrzak (Dept. of Anal. Chem., Inst. of Nuclear Res., Warsaw, Poland). *Chem. Anal., Warsaw*, 1960, **5** (6), 923–929.—*Procedure*—To the soln. of uranyl nitrate add 0·01M-tartaric acid (1 ml), 0·5% aq. gelatin soln. (2·5 ml) and N-KOH to pH 5·5 to 6·0. To this soln. add 10 ml of buffer soln. (N-dichloroacetic acid in 30% ethanol - N-Na acetate) (3:2), 10 ml of ethanol and 3 ml of phenylfluorone (**I**) soln. [75 mg of **I** dissolved in 210 ml of anhyd. ethanol and 12 ml of H_2SO_4 (1:6), and diluted with anhyd. ethanol to 250 ml]. Add H_2O to 50 ml and measure the extinction at 540 mμ. Compare with a calibration curve. The determination of 1 to 50 p.p.m. of Ti in uranyl nitrate is possible; the error is ±10%. B. K.

2818. **Analytical chemistry of zirconium. VII. Complexometric titration of zirconium and hafnium with Solochrome black 6BN as indicator.** J. Korkisch (Anal. Inst., Univ. Wien, Austria). *Z. anal. Chem.*, 1960, **178** (1), 42–46 (in German).—To the Zr or Hf soln. are added slowly 9 ml of 0·1N-HCl, 1 ml of Solochrome black 6BN (C.I. Mordant Black 3) (0·05% in methanol) as indicator, 1 ml of $AlCl_3.6H_2O$ soln. (40% in N-HCl) and 10 ml of methanol. The soln. is warmed on a boiling-water bath and titrated with 0·001M to 0·1M-EDTA (disodium salt) to a red end-point. This method is more susceptible to interference from foreign ions than that previously described (Korkisch and Farag, *Anal. Abstr.*, 1959, **6**, 3445) with Solochrome violet R (C.I. Mordant Violet 5) as indicator. The effects of acidity, temp., methanol and indicator concn. and foreign ions are discussed. For amounts between 1 and 100 mg the titration errors are ±2% and 0·2 to 2·5% for Zr and Hf, respectively, and, for amounts between 20 and 500 μg, ±5 to 6% and 2·5 to 10%, respectively.

B. B. BAUMINGER

2819. **X-ray fluorescence spectrometry. I. Application to the determination of thorium in minerals.** J. Bermúdez Polonio, F. de la Cruz Castillo and R. Fernández Cellini (Junta de Energia Nuclear, Madrid, Spain). *An. Real Soc. Esp. Fis. Quim., Madrid, B*, 1960, **56**, 569–578 (in Spanish).—An X-ray spectrometric method was developed for the determination of Th (0·01 to 0·5% of ThO_2) with Se as an internal standard. The concn. of Th is determined from a working curve prepared by plotting the percentage of ThO_2 against the ratio of the intensities of the Th L_{α_1} line to the Se K_{β_1} line. A Geiger counter is used together with associated electronic equipment for the measurement of intensities of X-ray emission lines. Tests were made to obtain preliminary information on the accuracy attainable by several instrumental factors. In addition, the statistics of X-ray emission process were checked in order to determine how experimental data agree with theory. The standard deviations are 3·5 and 2% for contents of 0·05 and 0·4% of ThO_2, respectively. NUCL. SCI. ABSTR.

2820. **Determination of metal ions by means of their oxalates. XI. Determination of thorium in the presence of zirconium.** Kyozo Kawagaki (Fac. of Engng, Niigata Univ., Nagaoka). *J. Chem. Soc. Japan, Pure Chem. Sect.*, 1960, **81** (6), 918–920.—Since Zr dissolves in an excess of oxalic acid soln., the determination of Th with oxalate (*Anal. Abstr.*, 1957, **4**, 2157) can be carried out in the presence of

Zr (< 50 mg per 50 mg of Th) by the use of 20 ml of 0·5N-oxalic acid in 200 ml of water.

K. SAITO

2821. **Estimation of aluminium in high-purity thorium compounds. An ion-exchange separation method.** V. T. Athavale and A. R. Subramanian (Atomic Energy Estab., Trombay, Bombay, India). *J. Sci. Ind. Res., India, B*, 1960, **19** (11), 431–432.—From 6 to 30 μg of Al can be separated from $\simeq$0·5 g of Th by quant. adsorption of Th on a column (30 cm × 1·5 cm) of a strongly basic anion-exchange resin (Merck III) at pH 2 to 2·5 and a flow-rate of 2 ml per min. The resin is washed with water acidified with H_2SO_4 (pH 2·1 ± 0·1) and the Al in the percolate (130 to 150 ml) is extracted into a 1% soln. of 8-hydroxyquinoline in $CHCl_3$, and then determined spectrophotometrically at 390 mμ against a blank of wash soln. plus ammonium acetate - acetic acid buffer (pH 4·9). Trace amounts of Fe, Co, Ni, Cu, Zn and Cd must be removed by the method of Margerum *et al.* (*Brit. Abstr. C*, 1953, 292); UO_2^{2+}, NO_3^- and >0·5 mg of F^- interfere. The error is from +1·2 to −4·9 μg (12 analyses).

W. J. BAKER

2822. **Measurement of the absolute concentration of thoron in the air of industrial premises.** Yu. N. Burmistenko. *Atomnaya Energiya*, 1960, **9** (6), 505–506.—The air is passed through an ionisation chamber at a constant rate, and the concn. of thoron (Tn) is calculated from the formula—

$$Q_o = \frac{i_{Tn} I_{Tn}}{V} \cdot \frac{1}{n} \text{ curies per litre,}$$

where Q_o is the concn. of Tn, i_{Tn} is the calibration coeff. for the chamber and equals $(Q_{Rn}/I_{Rn}) \times 1{\cdot}47$ curies per division per min. (where Q_{Rn} is the concn. of radon used in the stream of air for calibration of the chamber and I_{Rn} is the corresponding ionisation current), V is the volume of the chamber, and $1/n$ is the correction to the max. ionisation current for the given rate of flow of air ω. Curves are given relating n to ω for several values of V, the curves being calculated from the formula—

$$I = 5{\cdot}96 \times 10^{-14} a \frac{Q_o \omega}{\lambda} \left(1 - e^{-\lambda/^V\omega}\right),$$

taking lim. $I_{\omega \to \infty} = 1$; here I is the ionisation current, a is the efficiency of the chamber in counting the α-particles, and λ is the disintegration constant for Tn. To remove the influence of γ-radiation from Rn present in the air, the actual ionisation current is taken as the difference between that measured as described above, and the value obtained after stopping the flow of air and measuring the residual ionisation current after disintegration of the Rn in the chamber (5 to 6 min.).

C. D. KOPKIN

2823. **Spectroscopic isotope analysis of nitrogen by using a secondary electron multiplier.** G. Meier (Inst. f. Physikalische Stofftrennung, Leipzig, Germany). *Kernenergie*, 1960, **3**, 903–906 (in German).—A direct photometric method for the isotopic analysis of nitrogen is described. The intensity of the bandhead (0·1) of the $^{14}N_2$ and $^{14}N^{15}N$ molecules in the second positive system of nitrogen is measured by means of a photomultiplier. The advantage of the method is the short time required for an analysis and its independence of photographic plates. NUCL. SCI. ABSTR.

2824. **Polarographic reduction of hydrazoic acid and determination of azides.** J. Mašek (Polarographic Inst., Czech. Acad. Sci., Prague). *Coll. Czech. Chem. Commun.*, 1960, **25** (12), 3137–3141 (in English).—Hydrazoic acid in a strongly acid medium yields a reduction wave suitable for analytical purposes. The height of the limiting current is a linear function of the concn. of added azide from 1×10^{-4} to 6×10^{-3}M. The wave is best developed with 15M-H_2SO_4, but if nitrates are present 12M-H_2SO_4 must be used. At this concn. only nitrites interfere. During the preliminary removal of O with a stream of N a wash-bottle containing a soln. of the same concn. as the test soln. must precede the electrolytic cell. In all determinations it is necessary to add the same volume of test soln., because dilution of the H_2SO_4 causes a relatively large change in the diffusion coeff. of the depolariser.

A. J. BENNETT

2825. **Effect of chloride ions on the determination of nitrate by Leithe's method.** C. Heitner-Wirguin and D. Friedman (Dept. of Inorg. and Anal. Chem., Hebrew Univ., Jerusalem, Israel). *Talanta*, 1960, **7** (1–2), 121–123 (in English).—The determination of NO_3^- by reduction with Fe^{II} and subsequent titration of the excess of Fe^{II} is affected by the presence of Cl^-. The same concn. of Cl^- should be present in the blank and in the determination.

T. R. ANDREW

2826. **Photometric determination of phosphorus in the presence of large amounts of iron.** A. Almásy and I. Pallay (Univ., Veszprém, Hungary). *Nehézvegyip. Kut. Int. Közlem.*, 1960, **2** (1–2), 149–154.—The Fe is removed with cation-exchange resin (Dowex 50 or Wofatit F, treated with HCl). The soln. is treated with ammonium molybdate and reduced, and the P is determined photometrically as molybdenum blue.

J. G. GORODI

2827. **Alkalimetric determination of phosphoric oxide.** K. Kadič (Spolek Pro Chem. a Hutní Výr., Ústí nad Labem, Czechoslovakia). *Chem. Prûmysl*, 1960, **10** (12), 627–629.—The method is based on the reaction—$H_2PO_4^- + Ce^{3+} \rightarrow CePO_4 + 2H^+$; the liberated H^+ are titrated with NaOH soln. *Procedures:* (*i*) *With trisodium phosphate*—Dissolve the finely powdered sample (2 g) in H_2O (100 ml) and HCl (1:1) (10 ml) and dilute with H_2O to 250 ml. To 100 ml add H_2O (100 ml) and mixed indicator soln. (mix 0·1 g of bromocresol green and 0·02 g of methyl orange, then add 5 ml of 0·1N-NaOH, mix and dilute with H_2O to 100 ml) (0·6 ml) and titrate first with N-NaOH and then with 0·1N-NaOH till the colour turns bright green. Add $Ce(NO_3)_3$ soln. ($\simeq$5%) [dissolve 25 g of $Ce(NO_3)_3$ in 300 ml of H_2O, filter off the undissolved residue, add 15 drops of indicator soln., neutralise with N-NaOH, filter and if necessary adjust the acidity by adding HCl or NaOH soln. till the colour again turns bright green] (15 ml), mix and titrate the supernatant liquid with 0·1N-NaOH to the bright green end-point. (*ii*) *With crude phosphate*—Evaporate the powdered sample (1·5 g) with conc. HNO_3 (8 ml) and conc. HCl (12 ml) nearly to dryness on a sand bath, cool, dilute with H_2O (50 ml) and HCl (1:1) (10 ml) and boil. Cool, dilute to 250 ml with H_2O and pass 25 ml through a 20-cm column of Wofatit FN at 10 ml per min. to remove Fe and Al; wash the column with H_2O (5 × 25 ml) and titrate the percolate as described above. (*iii*) *With superphosphate*—Dissolve the sample (8 g) by a 30-min.

vigorous stirring in 800 ml of H_2O. Then dilute to 1 litre, filter through paper, and to 50 ml of filtrate add NaCl (1 g), H_2O (100 ml) and indicator and titrate as described above; SO_4^{2-} cause difficulties in the precise determination of the end-point. Chlorides, CO_3^{2-}, F^- and Ca do not interfere. The relative error is $\gtrsim \pm 1\%$. J. ZÝKA

2828. **Reduction of arsenic with liquid amalgam and differential titration of arsenic and antimony.** Chozo Yoshimura and Susumu Uno (Fac. of Engng, Kinki Univ., Fuse, Osaka). *J. Chem. Soc. Japan, Pure Chem. Sect.*, 1960, **81** (5), 736–738.—Both As^{III} and As^{V} are reduced within 30 min. with saturated zinc amalgam (150 g for $\simeq$10 mg of As) in 2N to 4N-HCl to produce AsH_3; this is passed into $AgNO_3$ soln. and the pptd. Ag is titrated with KI soln. in HNO_3, with Ce^{4+} - starch soln. as indicator. Although Sb^{V} is reduced to Sb^{III} with saturated tin amalgam in N-HCl at 60° within 5 min., As^{V} remains unchanged. Under the same conditions, but in the presence of Mo^{VI}, As^{V} is reduced to As^{III}. Since Mo^{VI} is simultaneously reduced to Mo^{III}, As can be differentially determined in the presence of Sb by titrating in the presence and absence of a known amount of Mo^{VI}. K. SAITO

2829. **Determination of vanadium as silver vanadate.** F. Maggio and F. P. Cavasino (Palermo Univ., Sicily). *Ann. Chim., Roma*, 1960, **50** (10), 1280–1289.—Examination of the effect of pH on the pptn. of vanadate as silver metavanadate ($Ag_2O.V_2O_5$) showed that a quantitative pptn. could not be obtained at pH <6, but the pptn. was satisfactory at pH 6·2 to 6·8. For the determination of vanadate as silver orthovanadate ($3Ag_2O.V_2O_5$), a soln. of NH_4VO_3 acidified with HNO_3 is treated with aq. NaOH (5%) until decolorised, and then, after heating for 1 min., until just alkaline to phenolphthalein. The soln. is diluted, cooled to $\simeq$40°, and treated first with conc. aq. NH_3 and then with an excess of aq. $AgNO_3$ (5%). The yellow ppt. first formed is transformed on boiling into a brown ppt. of Ag_3VO_4, which is finally filtered off, dried at 125°, and weighed. L. A. O'NEILL

2830. **Polarographic determination of niobium in highly alloyed steels.** D. J. Brindley [Anal. Development Sect., Joseph Lucas (Gas Turbine Equipment) Ltd., Shaftmoor Lane, Birmingham, England]. *Analyst*, 1960, **85**, 877–883.—The sample (1 g for Nb contents of 0·5 to 1·5%) is dissolved in HCl and HNO_3, the soln. is evaporated and the lightly baked residue is re-dissolved in HCl. The soln. is concentrated and treated with SO_2 and, after a procedure of dilution and boiling, the pptd. niobic acid (with other oxide impurities) is collected, ignited in a gold crucible at 800° and then fused with KOH at 300° to 450°. The melt is extracted with 2% aq. KOH and an aliquot of the extract is treated with EDTA soln., the pH is adjusted to 1·9, and the liquid is extracted twice with a soln. of 8-hydroxyquinoline in benzene, the organic layer, containing any Mo present, being discarded. Nitrogen is bubbled through the filtered aq. layer in a polarographic cell and the polarogram is recorded between −0·4 and −0·9 V. The height of the Nb wave is compared with that of a standard amount of Nb_2O_5 submitted to the same fusion and subsequent procedure as the sample. No interference is caused by Ta, W or Si. Treatment of the sample and standard must be identical. Quoted results indicate satisfactory accuracy. A. O. JONES

2831. **Determination of tantalum in high-tantalum ferro-alloys. [Radioactivation Analysis Symposium. Vienna, June 1959.]** D. Gibbons and H. Simpson (A.E.R.E., Harwell, England). *Pure and Appl. Chem.*, 1960, **1** (1), 135–137 (in English).—The sample is dissolved in HF and HNO_3 and some of the soln. (0·2 ml) is irradiated, together with a soln. containing a known weight of Ta, for 30 min. in a flux of 10^{12} neutrons per sq. cm per sec. If possible the concn. of Ta should be <2 mg per ml, so that the self-shielding error shall be <1%. After decaying for 24 hr. the activity due to ^{182}Ta is measured, preferably by γ-ray spectrometry, and the content of Ta is derived from the ratio of the activities of the standard and sample. G. J. HUNTER

2832. **Spectrographic determination of impurities in high-purity sulphur.** Z. G. Fratkin and I. Yu. Andreeva (All-Union Sci. Res. Inst. of Chem. Reagents, Leningrad). *Zavod. Lab.*, 1960, **26** (12), 1370–1371.—Determinations are carried out directly in an activated a.c. arc for Cu (0·0001 to 0·01%), Ag (0·0001 to 0·001%), Al (0·0005 to 0·01%), Fe (0·001 to 0·01%), Ni (0·0005 to 0·01%) and Co (0·0005 to 0·01%), and, after enrichment by ignition in the presence of SiO_2 and NaCl, for Cu (5×10^{-6} to 10^{-4}%), Ag (3×10^{-6} to 10^{-4}%), In (3×10^{-6} to 3×10^{-5}%), Ga (3×10^{-6} to 3×10^{-5}%), Ni (10^{-5} to 10^{-4}%) and Co (10^{-5} to 10^{-4}%). G. S. SMITH

2833. **Determination of traces of copper, lead and chloride in sulphuric acid by square-wave polarography.** Y. Yasumori (Yanagimoto Co., Ltd., Kyoto, Japan). *Coll. Czech. Chem. Commun.*, 1960, **25** (12), 3385–3386 (in English).—It was found experimentally that the optimum concn. of acid for the determination of Cu is 5M while for Pb and Cl^- it is 10M. After suitable dilution of the acid, O and SO_2 are removed by a stream of N. The sensitivity is increased by raising the temp., Cu and Pb being determined at 50° and Cl^- at 25°. A. J. BENNETT

2834. **Photometric end-point in the precipitation titration of sulphate.** U. Niwa and E. P. Parry (Union Res. Centre, Union Oil Co. of California, Brea, U.S.A.). *Chemist Analyst*, 1960, **49** (4), 102–105 (in English).—The titration of SO_4^{2-} with $Pb(NO_3)_2$ soln., with dithizone as indicator (*cf.* Archer, *Anal. Abstr.*, 1957, **4**, 2963), can be increased in sensitivity by detecting the end-point with a filter photometer at $\simeq$620 mμ. Samples containing 20 p.p.m. of SO_4^{2-} can be titrated directly if a period of 2 to 3 min. is allowed between the addition of aliquots of $Pb(NO_3)_2$ soln. The accuracy is $\simeq$2%. Chloride, in concn. up to 1680 p.p.m., has little effect, but a very considerable increase in titration equilibrium time is necessary with 3380 p.p.m.; ClO_4^- and H_2O_2 interfere and must be destroyed before titration. W. T. CARTER

2835. **Spectrographic determination of selenium in powdered samples.** O. F. Chesnokov and V. S. Sukhnevich. *Zavod. Lab.*, 1960, **26** (12), 1372.—Various lines in the spectrum excited by an a.c. arc are used to determine contents of Se from 0·003 to 5%. G. S. SMITH

2836. **Spectrophotometric determination of selenium(IV) with 1,1′-dianthrimide.** F. J. Langmyhr and S. H. Omang (Chem. Inst. A, Univ. of Oslo, Blindern, Norway). *Anal. Chim. Acta*, 1960,

23 (6), 565–568 (in English).—In conc. H_2SO_4, 1,1′-dianthrimide forms with Se^{IV} a 1:1 complex with absorption max. at 480 and 585 mμ. For photometry the colour is developed by heating the reaction mixture at 70° for 16 hr. and the method is suitable for the determination of Se in concn. up to 0·2 mg in 25 ml. The extinction is critically dependent upon the concn. of the H_2SO_4, and B^{III}, Ge^{IV}, Te^{IV} and Br^- interfere. H. N. S.

2837. **[Spectrographic] determination of traces of impurities in selenium.** Z. G. Fratkin and N. G. Polivanova (All-Union Sci. Res. Inst. of Chem. Reagents, Leningrad). *Zavod. Lab.*, 1960, **26** (12), 1372–1374.—An a.c. or d.c. arc is used for determining traces of Ag, Al, Bi, Cd, Co, Cu, Fe, Ga, Hg, In, Mn, Ni, Pb, Sb, Sn and Te without preliminary enrichment. G. S. SMITH

2838. **Spectrophotometric determination of chromium(III) with EDTA.** G. den Boef, W. J. de Jong, G. C. Krijn and H. Poppe (Lab. for Anal. Chem., Univ. of Amsterdam, Netherlands). *Anal. Chim. Acta*, 1960, **23** (6), 557–564 (in English).—The spectral characteristics of the Cr^{III} - EDTA complex are investigated under different pH conditions. Copper, Fe, Co and Ni also form coloured complexes and can interfere with the spectrophotometric determination of Cr with EDTA. Three procedures are described and the choice is determined by the other metals present. Procedure *A* is applicable in the presence of metals of groups 2 and 3, excepting Cu, Co and Ni. Iron is oxidised with Br, EDTA is added and its Cr complex is formed by heating. The soln. is neutralised with aq. NH_3 and buffered at pH 10 and the foreign metals are pptd. by heating with $CaCl_2$. The extinction due to the Cr^{III} - EDTA complex in the supernatant liquid is measured at 590 mμ or, after acidifying to pH 4·0, at 545 mμ. Procedure *B* is used when metals of groups 2 and 3, excepting Ni, are present (*cf.* Krijn and den Boef, *Anal. Abstr.*, 1961, **8**, 466). The soln. is neutralised with aq. NH_3 and is heated with EDTA. After buffering at pH 10, further heating with thioacetamide and $CaCl_2$ precipitates the interfering metals. The supernatant liquid is buffered at pH 10 and its extinction is determined at 590 mμ. Procedure *C* is used when Al, Fe^{III}, Zn, Mn, Co, Ni or Cu is present. The concn. of Co must not exceed that of Cr. In an aliquot of the soln. all the metals are complexed by heating with EDTA, the soln. is then buffered at pH 3·5 and the extinction due to all the metals is measured at 545 mμ. A second aliquot is buffered at pH 3·5, EDTA is added and the extinction due to the foreign metals at 545 mμ is measured immediately. Since the EDTA complex of Cr^{III} is formed very slowly at room temp., the difference between the two extinctions is a measure of the Cr present. H. N. S.

2839. **Back-titration with mercuric nitrate in alkaline medium. Determination of small amounts of chromium(III) and analysis of its binary mixtures with some other metals.** H. Khalifa and M. M. Khater (Fac. of Sci., Cairo Univ., Giza, Egypt). *Z. anal. Chem.*, 1961, **178** (4), 260–265 (in English).—Tervalent chromium (0·05 to 15 mg) is determined by addition of excess of 0·01M to 0·04M-EDTA (disodium salt), boiling the mixture for ⩽15 min., and potentiometric back-titration of the hot soln. with 0·01M to 0·05M-$Hg(NO_3)_2$ at pH 8·5 to 10·5. The titration apparatus is similar to that previously described (Khalifa, *Anal. Abstr.*, 1959, **6**, 48). The method was applied to the analysis of binary mixtures of Cr with Th, Cu^{II}, Bi or Hg^{II}, and the results are tabulated. B. B. BAUMINGER

2840. **Chemical studies of the ocean. LXXX. Quantitative determination of chromium in seaweed.** Yoshio Yamamoto (Chem. Dept., Kyoto Gakugei Univ., Fushimi-ku). *J. Chem. Soc. Japan, Pure Chem. Sect.*, 1960, **81** (3), 388–391.—The photometry of Cr with diphenylcarbazide (**I**) was modified for application to seaweed. The Cr (≃10 μg per 1 g of ash) is made insol. by ignition of the sample, and the ash is fused with $K_2S_2O_7$. The Cr is co-pptd. with Al [5% $Al_2(SO_4)_3$ soln.] (2 ml), and Fe and V are removed by extraction with cupferron in CCl_4 (5%) from 1·5N-H_2SO_4 soln. (≃50 ml). The aq. layer is treated with conc. HNO_3 to decompose the cupferron. Chromium is again pptd. with Al, dissolved in 1·3N-H_2SO_4 (≃50 ml), oxidised with Na_2BiO_3 (≃30 mg) and $AgNO_3$ soln. (10%) (3 drops) and determined photometrically with **I**.

K. SAITO

2841. **Determination of small amounts of molybdenum in tungsten and tungsten compounds.** H. Buss, H. W. Kohlschütter and S. Miedtank (Eduard Zintl-Inst. f. Anorg. und Phys. Chem., Tech. Hochsch., Darmstadt, Germany). *Z. anal. Chem.*, 1960, **178** (1), 1–11 (in German).—The procedures described permit 0·05 p.p.m. of Mo to be determined in ⩾50 g of sodium tungstate, ⩾10 g of calcium tungstate or ⩾10 g of tungsten metal. A scheme is given for the separation of Mo from these materials. Molybdenum is pptd. with thioacetamide under pressure in the presence of tartaric acid and potassium antimonate and the MoS_3 and Sb_2S_3 formed are filtered off and treated either with aqua regia or with Na_2O_2 in a micro Parr bomb. Hydrogen peroxide is boiled off completely, the soln. is neutralised with conc. HCl, and Mo is recovered either (*i*) as the green dithiol complex, which is extracted into $CHCl_3$ and its extinction measured at 665 mμ, or (*ii*) as the red thiocyanate complex, which is extracted into a mixture of CCl_4 and isoamyl alcohol and the extinction measured at 465 mμ. The content of Mo is found by reference to a calibration curve. The maximum error is 8% for concn. of 0 to 15 μg of Mo and 3% for concn. of 15 to 30 μg of Mo. Antimony does not interfere.

B. B. BAUMINGER

2842. **Extraction and determination of uranium as the dibenzoylmethane complex.** Tsunenobu Shigematsu and Masayuki Tabushi (Inst. Chem. Res., Kyoto Univ., Sakyo-ku). *J. Chem. Soc. Japan, Pure Chem. Sect.*, 1960, **81** (2), 265–269.—The photometric determination of U with dibenzoylmethane (**I**) (Yoe *et al.*, *Anal. Abstr.*, 1954, **1**, 80) was improved by extraction of the complex with butyl acetate (**II**) in the presence of the Ca - EDTA chelate. The optimum pH is 6 to 7 in the presence of EDTA soln. (10%) (2 ml) and $CaCl_2$ (equiv. to the EDTA), and the reaction between **I** (5% in acetone, 1 ml for <100 μg of U in 50 ml of water) and U is complete within 15 min. at 70°. The extinction of the **II** layer is proportional to the concn. of U for <10 p.p.m. There is significant interference from Cu (>2 mg per 0·1 mg of U), Mn (>2 mg), and Fe, Ti and Cr (>1 mg each). This method is applicable to the separation of U from fission products, the decontamination factor being 10^4, with the use of $Ce_2(SO_4)_3$ (≡1 mg of Ce) as hold-back carrier.

K. SAITO

2843. **Gravimetric determination of uranium in solutions obtained from ores by leaching with sulphuric acid.** T. Adamski and C. Deptuła (Dept. of Chem. Technol., Inst. of Nuclear Res., Warsaw, Poland). *Chem. Anal., Warsaw*, **1960**, **5** (6), 843–850.—*Procedure*—To 100 g of powdered ore add MnO_2 (1 g) and 8% H_2SO_4 (150 ml) and extract for 5 hr. at 80°. Filter off and wash the ppt. with 0·01N-H_2SO_4. Extract the filtrate with 0·1M-tri-n-octylamine soln. in light petroleum containing 3% (v/v) of n-octanol. Re-extract the U with M-NH_4NO_3 acidified with HNO_3. To an aliquot (≃50 mg of U) add 10% aq. EDTA soln. (10 ml), 20% aq. ammonium acetate soln. (25 ml) and aq. NH_3 to pH 5·1. Dilute the soln. to 180 ml, heat to 70° and precipitate U with 4% ethanolic 8-hydroxyquinoline soln. (10 ml). Collect the ppt. on a sintered glass filter, wash it with 0·01% 8-hydroxyquinoline soln. and dry it to constant wt. at 110°. The accuracy is ±0·9%. B. K.

2844. **Volumetric determination of uranium in the presence of high concentrations of iron.** T. L. Blalock (U.S. Dept. of the Interior, Bur. of Mines, Washington). *Rep. Invest. U.S. Bur. Min.*, **1960**, No. 5687. 9 pp.—To 75 ml of uranium soln. reduced in a Jones reductor are added 2 g of NH_4Cl, to prevent peptisation, 2 ml of $La(ClO_4)_3$ (=20 mg of La) as carrier, and 20 ml of 50% HF. The mixture is centrifuged and the UF_4 is collected and washed with 2% HF - 2% NH_4Cl soln. (2 × 15 ml), then dissolved in 84% H_3PO_4 (2 to 4 ml) and re-pptd. with HF. It is then fumed with 5 ml of 72% $HClO_4$ and the soln., with 8 ml of conc. HCl, is made up to 35 ml with H_2O and passed through a lead reductor before titration with standard $Ce(SO_4)_2$ soln. End-points are determined by visual and potentiometric methods, visual methods being preferred for low concn. of U (0·1 to 0·2 mg per 100 ml). Iron in amounts up to 500 mg is eliminated by this method. S.C.I. Abstr.

2845. **Photometric determination of uranium with arsenazo.** V. I. Kuznetsov and T. N. Kukisheva. *Zavod. Lab.*, 1960, **26** (12), 1344–1346.—The sample (containing 0·5 to 5 mg of U) is decomposed with mixed acids and evaporated to dryness. The residue is treated with 2 ml of HNO_3 and 20 ml of satd. $Ca(NO_3)_2$ soln. and heated for 2 to 3 min. After dilution to 50 ml with the $Ca(NO_3)_2$ soln., 5 or 10 ml of the decanted or filtered soln. in a graduated tube is mixed with 5 ml of EDTA (disodium salt) soln. [concn. not stated] and 5 to 10 ml of ethyl methyl ketone and well shaken. An aliquot (1 to 2 ml) of the extract is transferred to a 50-ml flask containing 25 ml of water, 2·0 ml of 0·1% arsenazo soln. and 2·0 ml of 25% hexamine soln. and then diluted with water to 50 ml. The extinction is measured with a red filter. The liquid in the cell is then mixed with six drops of 30% H_2O_2 and the extinction is measured again after 30 to 40 sec. The difference corresponds to the content of U; if it exceeds 0·050 the extraction is repeated. The method was tested on samples of ores, mixed oxides of Be and U, and Bi - Pb eutectic and was satisfactory over the concn. range 0·02 to 1·43%. G. S. Smith

2846. **Spectrophotometric determination of uranium and thorium with arsenazo.** H. P. Holcomb and J. H. Yoe (Pratt Trace Analysis Lab., Dept. of Chem., Univ. of Virginia, Charlottesville, U.S.A.). *Microchem. J.*, 1960, **4** (4), 463–480.—Arsenazo forms coloured complexes with Th and U. The colour due to the thorium complex alone (max. 565 mμ) appears at a pH <2, and U does not interfere if its concn. is <20 times that of Th. At pH 8·5 both complexes are formed. They do not have the same absorption maximum, but at the maximum for U (596 mμ) the absorptions are additive, and if the Th has been determined at pH 1 to 2 and 565 mμ its absorption at pH 8·5 and 596 mμ may be calculated from a calibration curve of the thorium complex. *Procedure*—Add $LiNO_3$ (8 g) to 10 ml of a soln. containing 2 to 7 mg of the nitrates of Th and U and N in HNO_3. Extract with mesityl oxide (13 ml), and wash the organic phase with a soln. saturated with $LiNO_3$ and N in HNO_3 (3 × 10 ml). Remove Th and U from the mesityl oxide by extraction with water (× 3), and dilute the aq. extracts to 100 ml. *Determination of Th*—Adjust an aliquot of the extract to a pH between 1·5 and 3, add 10 ml of a KCl - HCl buffer (pH 0·8) and 2 ml of $2·14 \times 10^{-3}$M-arsenazo. Dilute to 50 ml and measure the extinction at 565 mμ. *Determination of U*—Adjust an aliquot of the aq. extract to pH 2·5 to 5·5. Add $2·14 \times 10^{-3}$M-arsenazo (10 ml), mix and add buffer soln. (pH 8·6) (10 ml). Dilute to 50 ml and measure the extinction at 596 mμ against a reagent blank. The mesityl oxide extraction removes interfering ions except Zr and Fe, but good results are obtained if Zr + Fe is ⩾0·1(U + Th). The standard deviation for Th is 1·87% and for U 2·04%. A. J. Bennett

2847. **X-ray fluorescence spectrometry. II. Application to the determination of uranium in minerals.** J. Bermúdez Polonio, F. de la Cruz Castillo and R. Fernández Cellini (Junta de Energia Nuclear, Madrid, Spain). *An. Real Soc. Esp. Fis. Quím., Madrid, B*, **1960**, **56**, 579–588 (in Spanish).—In the method developed, Sr was found to be the most suitable internal standard. A Philips Norelco X-ray fluorescence spectrometer was used, equipped with a lithium fluoride crystal acting as a diffraction-grating analyser. The intensity of the U $L\alpha_1$ line is calculated and related to that of the corresponding Sr K_α line, both detected with a scintillation counter. Sources of error and interferences were investigated. The lower concentration limit is 0·01% of U_3O_8 and the standard deviations are 5·1 and 2·2% for contents of 0·1 and 0·5% of U_3O_8, respectively. A single determination can be made in ≃15 min. Nucl. Sci. Abstr.

2848. **Polarographic analysis of uranium. I. Polarographic behaviour in ammonium carbonate and sodium triphosphate.** Toshifusa Murata (Hitachi Res. Lab., Hitachi, Ibaraki-ken). *J. Chem. Soc. Japan, Pure Chem. Sect.*, 1960, **81** (3), 440–444.—Uranyl ions give two-step waves corresponding to $U^{VI} \rightarrow U^{V}$ and $U^{V} \rightarrow U^{IV}$ in ammonium carbonate and in sodium triphosphate (**I**) soln., the $E_{\frac{1}{2}}$ value and the wave height depending on the concn. of the electrolyte. The first wave is not reversible, but has a distinctive shape with a good reproducibility. In 0·1M-**I** containing 0·1M-ammonium carbonate (pH 9·6) it has a kinetic nature and the wave height at −1·10 V *vs.* the S.C.E. is proportional to the concn. of U (0·1 to 1 millimole). There is no interference from Mo, Cr, Co, Mn, Zn, V, Cu, Fe, Cd, Pb or Zr. The wave of Ni overlaps the first wave. K. Saito

2849. **Determination of uranium and plutonium in uranium - plutonium - fission element alloys.** R. P. Larsen and C. A. Seils, jun. (Chem. Engng Div., Argonne Nat. Lab., 9700 South Cass Ave.

Argonne, Ill.). *Anal. Chem.*, 1960, **32** (13), 1863–1866.—A tri-n-butyl phosphate - HCl system is used to separate U and Pu from certain fission elements and from each other prior to X-ray spectrographic determination of U and spectrophotometric determination of Pu as the nitrate complex. Co-precipitation of PuF_3 with LaF_3 has also been used for the separation. D. C. ARMSWORTH

2850. **Separation and determination of plutonium in uranium - fission product mixtures.** W. J. Maeck, G. L. Booman, M. E. Kussy and J. E. Rein (Atomic Energy Div., Phillips Petroleum Co., Idaho Falls). *Anal. Chem.*, 1960, **32** (13), 1874–1876.—*Procedure*—Add 6 ml of $Al(NO_3)_3$ salting soln. (Maeck *et al.*, *Ibid.*, 1960, **32**, 605) to 0·1 ml of 0·05M-$KMnO_4$, then add 1 ml of sample soln. and 3 ml of isobutyl methyl ketone, extract for 5 min., and centrifuge. Transfer 3 ml of 3·125M-HNO_3, 2 ml of 1·25M-hydroxyammonium chloride and 2 ml of the organic phase to a tube and extract for 10 min., then centrifuge. Transfer 2 ml of the aq. phase to a tube containing 0·1 ml of fresh 0·2M-$FeSO_4$ and set aside for 5 min. Add 3 ml of fresh 0·22M-$NaNO_2$ and set aside until no more gas is evolved. Add 5 ml of 0·5M-thenoyltrifluoroacetone in xylene and stir for 20 min. Remove an aliquot of the organic phase, dry it on a planchet, ignite and measure the α-activity. D. C. ARMSWORTH

2851. **Spectrographic analysis of plutonium.** J. Artaud, M. Chaput and J. Robichet (Comm. a l'Energie Atomique, Centre d'Études Nucl., Saclay, France). *U.S. Atomic Energy Comm.*, Rep. CEA-1541, 1960. 47 pp.—Methods for the spectrographic determination of impurities in plutonium are considered. Applications of the copper spark method, sparking on graphite, and fractional distillation in the arc are described.

NUCL. SCI. ABSTR.

2852. **Determination of halides with the chromium(VI) - concentrated phosphoric acid reagent.** Toshiyasu Kiba and Ikuko Akaza (Chem. Dept., Fac. of Sci., Kanazawa Univ.). *J. Chem. Soc. Japan, Pure Chem. Sect.*, 1960, **81** (3), 449–451.—When chlorides are decomposed with $K_2Cr_2O_7$ in conc. phosphoric acid (**I**) (10% w/w) (prepared at 300°) a small part of the Cl is converted into CrO_2Cl_2. By the addition of $Ag_2Cr_2O_7$ (0·5% in **I**), all the Cl is evolved as gaseous Cl, which is absorbed in 1/30N-Na_3AsO_3 and titrated with 0·02N-iodine. There is no interference from SO_4^{2-}; NO_3^- produce nitrosyl chloride and so give low results. The sample soln. is evaporated to dryness with KOH soln. (10%) (2 ml) and treated with the reagent. On treatment with $K_2Cr_2O_7$ in **I**, bromides give only Br, which is similarly titrated. Iodides are oxidised to IO_3^-; after evolution of O ceases, activated charcoal (0·01 g) is added to the **I** layer, which is heated in a current of CO_2 for 15 min. Iodate is thus converted into iodine, which is similarly determined volumetrically. K. SAITO

2853. **Selective precipitation of silver halides from homogeneous solution. Separation of iodide, bromide and chloride using volatilisation of ammonia.** F. H. Firsching (Dept. of Chem., Univ. of Georgia, Athens, U.S.A.). *Anal. Chem.*, 1960, **32** (13), 1876–1878.—The silver halides are successively pptd. at decreasing pH. *Procedure*—Dissolve the sample (≃0·2 millimole) in 20 ml of H_2O and dilute to 200 ml with conc. aq. NH_3. Add the precipitant (0·4 millimole of $AgNO_3$ in 100 ml of conc. aq. NH_3) and thymolphthalein soln. and warm on a low-temp. hot-plate until the blue colour fades noticeably (≃2 hr.). When the pH at room temp. is 9·8 to 10·2, filter off the AgI. To the filtrate and washings add $AgNO_3$ soln. (pH ≮10), in amount approx. equivalent to the suspected Br^- content, and phenolphthalein soln. and warm on the hot-plate until the pink colour is completely discharged (≃1 hr.). When the pH at room temp. is 8·6 to 8·7, filter off the AgBr. To the filtrate and washings add $AgNO_3$ soln. (pH ≮8·7) in amount approx. equivalent to the suspected Cl^- content, warm on the hot-plate till the pH is <7·3 and filter off the AgCl. The errors are similar to those of the potentiometric method of Martin (*Anal. Abstr.*, 1958, **5**, 2196). R. E. E.

2854. **The formation of interhalogens.** E. Schulek and K. Burger (Inst. of Inorg. and Anal. Chem., L. Eötvös Univ., Budapest). *Talanta*, 1960, **7** (1–2), 41–45 (in English).—Evidence is produced for the formation of interhalogen compounds by the reaction between iodine or Br and $HgCl_2$ or AgCl. Free Br in a mixture of Br and BrCl may be determined by measuring the total oxidising power by the liberation of iodine from iodide, and treatment of a second aliquot with an excess of Cl, destruction of the excess with KCN and liberation of iodine from iodide. The difference in the amounts of iodine liberated is a measure of the original Br content. A procedure for the preparation of BrCl soln. is described. T. R. ANDREW

2855. **Determination of fluorine by alkaline hydrolysis of uranium and thorium fluorides.** M. Barrachina Gómez and L. Gascó Sánchez (Junta de Energia Nuclear, Madrid, Spain). *An. Real Soc. Esp. Fis. Quim., Madrid, B*, 1960, **56**, 589–596 (in Spanish).—The alkaline hydrolysis of the fluorides of uranium and thorium is studied, and a new method for the determination of the fluoride, based on an indirect volumetric titration with standard NaOH soln., is proposed. The compounds that may influence the hydrolysis of the uranium fluoride and that may be occasionally found in it as impurities are also studied. The method can be applied to uranium fluoride except when there is a large amount of UO_2F_2 or UO_3 present in the sample. The method is always applicable to thorium fluoride.

NUCL. SCI. ABSTR.

2856. **Determination of impurities in liquid chlorine.** T. De Vries and C. P. Savariar (Dept. of Chem., Purdue Univ., Lafayette, Ind., U.S.A.). *J. Amer. Wat. Wks Ass.*, 1960, **52** (10), 1338–1340.—Among methods for the determination of impurities in liquid chlorine, the pyridine method for chloroform determination is not specific and the chromic acid method to determine hydrochloric acid is troublesome. Measurements of residual gas volumes after absorption of chlorine by mercury and the determination of iron chloride in residues obtained by evaporation of chlorine are satisfactory, as is a method of determining nitrogen trichloride. A method for the determination of $SiCl_4$ is described. It is based on the production of molybdenum blue, with 1-amino-2-naphthol-4-sulphonic acid as reducing agent in the presence of tartaric acid, and measurement of the extinction at 815 mμ.

B. F. FULLAN

2857. **Spectrophotometric determination of chlorate impurities in ammonium perchlorate. Determination of reducing and oxidising impurities in hydrochloric acid.** E. A. Burns (Propulsion

Dept., Poulter Lab., Stanford Res. Inst., Menlo Park, Calif.). *Anal. Chem.*, 1960, **32** (13), 1800–1803.—The method is based on the reaction of ClO_3^- with benzidine in HCl medium to yield a coloured complex. *Procedure*—To the sample (6 g) dissolved in H_2O add benzidine soln. (2 g of benzidine plus 2·5 ml of conc. HCl made up to 500 ml with H_2O) (5 ml) and make up to 100 ml with HCl (1:1). Mix, and after 12 to 15 min. at room temp. measure the extinction at 438 mμ in a 1-cm Corex cell against a reference soln. [3 ml of benzidine soln. plus 97 ml of HCl (1:1)]. Follow a similar procedure with a soln. prepared by mixing 5 ml of 10^{-4}M-$KClO_3$ and 3 ml of benzidine soln. and diluting to 100 ml with HCl (1:1). The percentage of ammonium chlorate in the sample is determined from these readings by an equation which includes a correction for the amount of ammonium bromate in the sample. The graph of extinction *vs.* concn. of ClO_3^- does not pass through the origin. This is due to the presence of reducing (SO_3^{2-}) or oxidising (free Cl) substances in the HCl, which react with the benzidine. Equations are given for calculating the concn. of such impurities from a single measurement with a known concn. of $KClO_3$. R. E. E.

2858. **Catalytic determination of microgram amounts of iodide by amperometric titration.** K. Czarnecki (Dept. of Inorg. Chem., Univ., Łódź, Poland). *Chem. Anal., Warsaw*, 1960, **5** (6), 875–880.—Potassium iodide (0·12 to 0·76 μg) was determined by its catalysis of the H_3AsO_3 - Ce^{4+} reaction. The measurement of reaction rate as a function of I^- concn. was performed by amperometry without applied e.m.f.; Cl^-, Br^- and IO_3^- do not interfere in concn. up to 10^3 times that of I^-. The accuracy is ±2·5%. B. K.

2859. **Chelatometric titration of manganese in ferromanganese.** A. G. C. Morris (Ferralloys Ltd., Cato Ridge, Natal, S. Africa). *Chemist Analyst*, 1960, **49** (4), 105–106 (in English).—The method of Přibil and Kopanica (*Anal. Abstr.*, 1960, **7**, 997) is slightly modified to increase the titration volume. For 15 determinations on a sample containing 77·95% of Mn the modified method gave a mean Mn content of 77·93% and a standard deviation of ±0·21; 18 determinations by a second analyst on the same sample gave 78·08% and ±0·15. W. T. Carter

2860. **Chelatometric titration of manganese in ferromanganese.** K. Studlar (Res. Inst., V.I. Lenin Works, Plzen, Czechoslovakia). *Chemist Analyst*, 1960, **49** (4), 106–107 (in English).—The method is that of Přibil and Kopanica (*Anal. Abstr.*, 1960, **7**, 997), in which an improvement in accuracy is obtained by replacing 0·05M-EDTA with 0·025M-EDTA. In replicate determinations the standard deviation of a single determination is 0·06% of Mn. W. T. Carter

2861. **Spectrophotometric determination of manganese(II) with benzohydroxamic acid.** D. O. Miller and J. H. Yoe (Pratt Trace Analysis Lab., Univ. of Virginia, Charlottesville, U.S.A.). *Talanta*, 1960, **7** (1–2), 107–116 (in English).—In ammoniacal soln. (0·07 to 7M), Mn^{II} reacts with benzohydroxamic acid in aq. soln. or in dimethylformamide to give a coloured complex of Mn^{III} and benzohydroxamic acid having max. extinctions at 360 and 500 mμ. At 360 mμ the reagent and many metal ions interfere, while at 500 mμ only Fe^{II}, Fe^{III}, Co^{II} and Cu^{II} interfere, and these may be separated by passage of the soln. in 12N-HCl through Dowex 1-X8 anion-exchange resin and elution of the Mn with 6N-HCl. Magnesium retards the rate of reaction, but 98% of the value in the absence of Mg is found after 10 min. Beer's law is obeyed up to 10 p.p.m. of Mn and an extinction of 0·1 is given by 1·5 p.p.m. of Mn in a 1-cm cell at 500 mμ. Results for NBS samples of steel, bronze and magnesium alloy are in close agreement with NBS values. T. R. Andrew

2862. **Co-precipitation of permanganate ions with lead sulphate.** Kenjiro Hayashi and Yasumitsu Uzumasa (Fac. of Sci., Hokkaido Univ., Sapporo). *J. Chem. Soc. Japan, Pure Chem. Sect.*, 1960, **81** (6), 906–912.—The amount of co-pptd. MnO_4^- increases with increase in concn. of MnO_4^- (*e.g.*, 1×10^{-5} mole per 1 g of $PbSO_4$ in 0·001M-$KMnO_4$; 4×10^{-5} mole in 0·004M soln.) and with decrease in temp. With increase in excess of Pb, the amount of co-pptd. MnO_4^- decreases. When $PbSO_4$ is pptd. in the presence of excess of H_2SO_4, the amount of co-pptd. MnO_4^- increases with increase in amount of KNO_3 and $NaNO_3$ in the soln. The co-pptd. MnO_4^- do not react with oxalic acid in H_2SO_4. K. Saito

2863. **Liquid - liquid extraction of iron(III) with tributyl phosphate.** S. K. Majumdar and A. K. De (Jadavpur Univ., Calcutta, India). *Talanta*, 1960, **7** (1–2), 1–6 (in English).—Ferric iron may be extracted quant. into tributyl phosphate from Cl^- soln., 2M to 6M in HCl, with partition coeff. of 30 to 70. The results and conclusions of Specker and Cremer (*Z. anal. Chem.*, 1959, **167**, 110) are confirmed. T. R. Andrew

2864. **Extraction and flame-photometric determination of iron.** O. Menis and T. C. Rains (Anal. Chem. Div., Oak Ridge Nat. Lab., Tenn.). *Anal. Chem.*, 1960, **32** (13), 1837–1841.—Iron is extracted from soln. in 6M-HCl with isobutyl methyl ketone, and the emission of the Fe in the organic phase is measured at 372 mμ. Calibration curves are linear over the range 1 to 50 μg per ml, and the coeff. of variation ranges from 2 to 4%, according to the nature of the sample. Of 15 cations tested, only Cr^{VI}, Mo and Sn interfered at concn. >150 times that of the Fe; F^-, SO_4^{2-} and PO_4^{3-} did not interfere in molar concn., but 0·1M-NO_3^- and 0·1M-ClO_4^- quenched the emission of the Fe. Optimum conditions are described for the extraction of the Fe and the elimination of interference, and procedures are given for the determination of Fe in thorium dioxide, uranyl sulphate and copper sulphate soln. and various alloys. R. E. E.

2865. **Amperometric cerimetric titration of iron(II) with ferroin as indicator.** I. M. Kolthoff and B. B. Bhatia (School of Chem., Univ. Minnesota, Minneapolis, U.S.A.). *Microchem. J.*, 1960, **4** (4), 451–457.—Ferroin is used as an amperometric indicator because the reaction between Ce^{IV} and ferroin is extremely rapid, and at 0·65 V (*vs.* the S.C.E.) in N-H_2SO_4 a diffusion current proportional to the concn. of ferroin is obtained. A rotating platinum electrode of 2 sq. cm area is used against a S.C.E. To 50 ml of ferrous iron soln. ($>5 \times 10^{-5}$M-Fe^{2+}) is added 1 ml of 0·01M-ferroin. A potential of 0·65 V is applied and titration with standard $Ce(SO_4)_2$ soln. is carried out. An indicator blank is determined. Oxygen and As^{3+} do not interfere. The accuracy is within 1%. A. J. Bennett

2866. **Spectrophotometric determination of iron(III) with dibenzoylmethane.** Tsunenobu Shigematsu and Masayuki Tabushi (Inst. Chem. Res., Kyoto Univ., Sakyo-ku). *J. Chem. Soc. Japan, Pure Chem. Sect.*, 1960, **81** (2), 262–265.—Tervalent Fe forms a red complex with dibenzoylmethane (**I**) in a molar ratio of 1 to 3, which is extracted with butyl acetate (**II**) from an aq. soln. of pH 2·5 to 3·0. In **II** the complex has absorption max. at 343 and 410 mμ, whilst **I** has a marked absorption at 343 mμ. The extinction at 410 mμ is proportional to the concn. of Fe for <2·0 p.p.m. per 20 ml of **II**. Excess of **I** in acetone (5%, 0·5 ml per 50 ml of water) does not cause interference. The reaction between **I** and Fe^{III} is rather slow in water, but is complete within 10 min. at 70°; it is masked with EDTA. Copper (>20 μg per 20 μg of Fe), Cr (>100 μg), Mo and Ti give a positive error.

K. Saito

2867. **Potentiometric determination of boron in steel and alloys.** P. Ya. Yakovlev and G. V. Kozina (I. P. Bardin Central Sci. Res. Inst. of Ferrous Metallurgy). *Zavod. Lab.*, 1960, **26** (12), 1342–1343.—The sample (1 g) is dissolved under reflux in 30 ml of HCl (1:1) and the soln. is oxidised by boiling with 10 to 20 ml of 3% H_2O_2 soln. for 10 min. Any insoluble matter is filtered off and fused with Na_2CO_3 and the soln. of the melt in dil. HCl is added to the main soln. The soln. is diluted to 350 to 400 ml and 20% NaOH soln. (10 to 15 ml in excess) is added to precipitate hydroxides of Fe, etc. An aliquot (250 ml) of the filtered and diluted soln. (500 ml) is treated with 5% citric acid soln. (5 to 6 ml if the alloy contains 10% of Al) and made just acid to methyl orange by addition of HCl (1:1). The soln. is boiled for 5 min. to remove CO_2; no detectable loss of boric acid occurs during this operation. The cooled soln. is titrated potentiometrically to pH 6·9 with 2% NaOH soln. followed by 0·02N-NaOH. The boric acid is determined by the addition of 30 ml of invert sugar soln. followed by titration to pH 6·9 with 0·02N-NaOH.

G. S. Smith

2868. **Colorimetric determination of trace amounts of aluminium in steel.** D. Blair, K. Power, D. L. Griffiths and J. H. Wood (North Western Div., National Coal Board, Manchester, England). *Talanta*, 1960, **7** (1–2), 80–89 (in English).—A study of methods for the separation of Fe (0·5 g) from Al (25 to 2500 μg) led to the adoption of mercury-cathode electrolysis, followed by extraction of the cupferron complexes with $CHCl_3$. Aluminium is determined by the method of Scholes (*Analyst*, 1958, **83**, 615) with a nitrated Solochrome cyanine R (C.I. Mordant Blue 3) reagent (prep. described).

T. R. Andrew

2869. **Rapid method for the gravimetric determination of silicon in steel and cast iron by precipitation of the blue molybdosilicate complex of quinoline.** D. C. Filipov. *Compt. Rend. Acad. Bulg. Sci.*, 1960, **13** (5), 543–546 (in French).—The yellow molybdosilicate - quinoline complex used for determining Si in aluminium alloys and aluminosilicates cannot be applied to the determination of Si in ferrous materials because of interference from P, As and V in oxidising media. The difficulty is overcome by pptn. of the blue molybdosilicate with quinoline in a reduced soln., as follows. The sample (0·25 g) is dissolved in 18 ml of 5% H_2SO_4, and filtered, the residue being washed with warm water. The filtrate (≃100 ml) is cooled to 20°, 20 ml of 5% ammonium molybdate soln. is added and the mixture is set aside for 5 to 10 min. After adding 10 ml of HCl and 30 ml of 2% quinoline reagent, with stirring, the mixture is heated to 70° to 80° and then cooled to 20°. The ppt. is collected in a weighed Gooch-type crucible and washed (× 6) with quinoline wash soln., then dried at 150° to constant weight. Examples quoted show that 0·16 to 2·45% of Si can be determined with good reproducibility. For the reagent soln., 20 ml of quinoline is dissolved in 50 ml of HCl, the soln. is diluted with an equal volume of water, stirred and made up to 1 litre with water. For the wash soln., 25 ml of reagent soln. is diluted to 1 litre.

R. Presser

2870. **Comparison of the completeness of recovery of the sulphur contents of foundry products by various combustion processes.** D. Blazejak-Ditges. *Arch. Eisenhüttenw.*, 1960, **31** (12), 717–722.—Comparative tests by the methods of Holthaus (*Stahl u. Eisen*, 1924, **44**, 1514; *Arch. Eisenhüttenw.*, 1931–1932, **5**, 95), Pontet and Boulin (*Anal. Abstr.*, 1954, **1**, 1497) and Koch *et al.* (*Arch. Eisenhüttenw.*, 1958, **29**, 543) show that the O used should be freed from impurities by a pre-combustion; it is then drawn through the main furnace by a water-jet pump at a rate sufficient to ensure complete combustion, but allowing complete absorption of SO_3 in the aq. H_2O_2. The H_2SO_4 is then determined by titration with 0·025N-NaOH without empirical standardisation.

A. R. Pearson

2871. **Activation analysis of minor constituents in high-alloy steels.** [**Radioactivation Analysis Symposium. Vienna, June 1959.**] J. Hoste (Lab. of Anal. Chem., Univ. of Ghent, Belgium). *Pure and Appl. Chem.*, 1960, **1** (1), 99–110 (in English).—A procedure involving the use of an internal standard has been developed; two quantities of the material to be analysed are irradiated, but with one is mixed homogeneously a known amount of the element to be determined. The determinations of V and of W in steel are considered in detail.

G. J. Hunter

2872. **Spectrographic analysis of non-metallic inclusions.** M. Jelínek and M. Mandl (V.Ú.Ch.Z. Prague). *Hutn. Listy*, 1960, **15** (12), 979–982.—A soln. of Co, which is not present in the tested samples, was used as the internal standard and a rotating graphite disc as the electrode. Two methods were used for the dissolving and preparation of the sample. (*i*)—Fuse the sample with a 40-fold excess of $Na_2B_4O_7$ (in the presence of Cr add a crystal of $NaNO_3$) in a platinum crucible. Cool, add standard $CoCl_2$ soln. and citric acid so that the ratio of Co to inclusions is 10:1, and dissolve by heating the crucible in an aluminium block at 80°. (*ii*)—Fuse the sample with a 40-fold excess of a mixture of anhyd. Na_2CO_3 and $Na_2B_4O_7$ (3:2), and dissolve the melt in a mixture of $CoCl_2$, citric acid and HCl. The error is ±6%.

J. Žýka

2873. **Effect of aluminium nitride on the determination of oxide inclusions in stabilised Thomas steel.** E. Piper, H. Hagedorn, and M. Fröhlich (Röchling'schen Eisen u. Stahlwerke, Völklingen, Germany). *Arch. Eisenhüttenw.*, 1960, **31** (10), 577–585.—The steel contains Al as AlN crystals, which are recovered with the oxide inclusions by anodic treatment. If the Al content of the steel is high (0·1 to 0·4%) the crystals are coarse (15 to 40 μ) and resist chlorination at 300° or treatment with aq. NaOH (5%) at <100°. Such steels must be reheated at 1300° for <1 hr. and water-quenched

to ensure that the AlN is in finely crystalline form and can be destroyed by treating the anode residue with aq. NaOH at 60° for 30 min. The possibility of determining the NH_3 so produced is mentioned.
A. R. PEARSON

2874. **Single quantitative chemical determinations of non-ferrous metals used in iron and steel alloys. I.** A. Klinkmann. *Metall*, 1960, **14** (12), 1190–1194.—Details are given for the volumetric determination of Mn by the persulphate - sodium arsenite (separation of alloying elements with ZnO) and Volhard methods. The photometric determination of Cu as a cuprammonium compound, the rapid determination of Si (0 to 1%) and Cr (0 to 5%) in the absence of W by attack and oxidation with $HClO_4$, and the volumetric determination of Cr (0 to 30%) and V (>1%) in acid-soluble steels by the persulphate method, with both visual and potentiometric indication of the end-point, are described. J. H. WATON

2875. **Spectrographic analysis of ferro-alloys and chromium for the content of non-ferrous metal impurities.** S. B. Shubina, A. B. Shaevich and E. P. Basova (Ural Sci. Res. Inst. of Ferrous Metals). *Zavod. Lab.*, 1960, **26** (12), 1364–1366.—The use of arc excitation for determining low concn. (5×10^{-5} to 10^{-2}%) of Pb, Sn, Sb, Bi, As, Zn and Cd in ferro-alloys and chromium is described.
G. S. SMITH

2876. **Photo-electric spectrographic analysis of ferro-chrome and ferro-tungsten slags.** A. K. Tumanov (Chelyabinsk Ferro-alloy Works). *Zavod. Lab.*, 1960, **26** (12), 1366–1368.—Analysis is carried out by use of the lines W 4008·75 A and the triplet Cr 5208·44, 5206·04 and 5204·52 A and photo-multipliers. G. S. SMITH

2877. **The flame-photometric characteristics of the transition elements.** E. Pungor and I. Konkoly-Thege (L. Eötvös Sci. Univ., Budapest). *Magyar Kém. Foly.*, 1960, **66** (12), 520–522.—Soln. of cobalt and nickel salts in water or acid were prepared for investigation by the addition of known amounts of H_2SO_4 and ethanol. The effects of flame temp., speed of atomisation, and SO_4^{2-} and ethanol concn. on the atomic and molecular band spectra of the Co and Ni were studied, as were the effects of the alkali metals. A Beckman DU spectrophotometer with a N - H - O flame was employed. It is considered that the determination of the transition elements by flame photometry is possible only in exceptional circumstances. B. VASZKO

2878. **Determination of heavy metals with EDTA. IV. Separation of cobalt from various heavy metals.** Susumu Takamoto (Fac. of Sci., Gakushu-in Univ., Toshima-ku, Tokyo). *J. Chem. Soc. Japan, Pure Chem. Sect.*, 1960, **81** (3), 457–460.—When Co (0·2 to 5 mg) is pptd. as $K_3Co(NO_2)_6$, co-pptn. of weakly basic metals such as Sn, Sb, Fe, Cr and Al (50 to 120 mg) is avoided by the addition of lactic acid (75%, <1·5 ml per 10 ml of soln.), which is superior to tartaric acid. By the use of $NaNO_2$ as well as KNO_3 (40 g of KNO_3 and 20 g of $NaNO_2$ in 20 ml of water), the solubility of the ppt. is reduced. Digestion of the reactants at 80° for 1 hr. followed by cooling for 1 hr. with water suffices for the quant. pptn. of Co. Titanium, Bi and Cu are masked with *NN*-di(hydroxyethyl)glycine (25% soln., 1 ml per 10 ml); Ni and Pb (≃100 mg each) are removed by re-pptn. The centrifuged ppt. is dissolved in HCl and titrated with EDTA in the presence of KSCN, ammonium acetate and acetone. K. SAITO

2879. **Macro- and micro-determination of traces of cobalt. Spectrophotometric and polarographic determination of cobalt in MA 2 B Edelweiss steel.** D. Monnier, W. Haerdi and J. Vogel (Univ. Genève, Switzerland). *Anal. Chim. Acta*, 1960, **23** (6), 577–584 (in French).—In the macro-method the sample (0·1 to 0·5 g) is dissolved in aqua regia and ^{60}Co is added to enable manipulative losses to be determined. The soln., diluted with 9N-HCl, is extracted with ethyl ether to remove the greater part of the Fe. On passing the soln. through Dowex 1-X8 ion-exchange resin, Ni and Cr pass through the column, Co is eluted with 4N-HCl and Fe is retained. The eluate is adjusted to pH 8·0 and Co is extracted with an ethereal soln. of dithizone. The Co - dithizone complex is destroyed by heating with $HClO_4$ and the Co is determined spectrophotometrically with nitroso-R salt. The chemical yield is determined in the final soln. by measuring the γ-activity and is generally 80 to 85%. For a 100-mg sample the error may amount to 4% for Co contents of ≃0·04%. Amounts down to 0·02 μg of Co can be determined. In an adaptation to the micro scale (1 to 10-mg samples) the manipulation is simplified, the possible error increases to 8% for Co contents of ≃0·04%, and 0·005 μg of Co can be determined. The polarographic method is applied to the appropriate fraction of the eluate from the ion-exchange column. When >1 μg of Co is determined the error is <10%. H. N. S.

2880. **New reactions for the detection and quantitative photometric determination of small amounts of nickel.** H. Hartkamp (Inst. f. Spektrochem. und angew. Spektroskopie, Dortmund-Aplerbeck, Germany). *Z. anal. Chem.*, 1960, **178** (1), 19–27 (in German).—The colorimetric determination of Ni in 10 to 500-μg amounts is described. The oxidation of Ni^{2+} to Ni^{4+} is effected by IO_4^- and other oxidising agents, and the Ni^{4+} yield coloured complexes with pyridine-2,6-diacetaldoxime (**I**) and phenyl 2-pyridyl ketoxime (**II**). *Procedure*—To a soln. containing 10 to 500 μg of Ni (pH 7 to 9·5) are added 10 ml of **I** or **II** (prep. described), 10 ml of buffer soln. (10 ml of 2N-aq. NH_3 and 900 ml of 2N-NH_4Cl) and 10 ml of satd. KIO_4 soln. The soln. is warmed for a few seconds, cooled, diluted with water to 100 ml, and the blue Ni complex obtained with **I** or the greyish-blue complex obtained with **II** is measured at 585 mμ or 575 mμ, respectively. The content of Ni is read from a calibration curve; the effects of interfering cations are tabulated.
B. B. BAUMINGER

2881. **Semi-micro determination of nickel with 4-methylnioxime [4-methylcyclohexane-1,2-dione dioxime].** R. E. Longo (Com. Iberoamer. de Normalizaciones, Inst. Invest. Microquím., Rosario, Argentina). *Microchem. J.*, 1960, **4** (4), 527–530.—Adjust the test soln. to pH 1·0 and heat to 60° with 2 ml of saturated 4-methylnioxime soln. for each mg of Ni present. Add aq. NH_3 until a pink turbidity appears. Add slowly a vol. of 20% ammonium acetate soln. equal to 10% of the total vol. Set aside for 30 to 45 min. at 60°, filter, wash the ppt. and dry it at 120°. The conversion factor is 0·1590. With 2 to 10 mg of Ni the mean error for nine determinations was 0·86%.
A. J. BENNETT

2882. **New method for the preparation of furil α-dioxime and its application to the amperometric titration of nickel.** Koji Ishikawa (Chem. Dept., Maritime Tech. College, Ashiya, Hyogo-ken). *J. Chem. Soc. Japan, Pure Chem. Sect.*, 1960, **81** (3), 460–462.—To prepare pure furil α-dioxime (**I**), the red complex of **I** with Ni is decomposed with HCl and ethanol. The complex is obtained by treating furil in ethanolic KOH soln. with hydroxyammonium chloride, filtering off the KCl and adding $NiSO_4$ and ammoniacal NH_4Cl soln. The amperometric titration of Ni (≃1 mg) with **I** in 50% aq. ethanol is effected in an ammoniacal buffer of pH 9 to 10 at −1·70 V *vs.* the S.C.E. The pptn. of the Ni - **I** complex is retarded in the presence of gelatin. The error is <3%. K. SAITO

2883. **Determination of nickel and manganese with formaldoxime.** A. Okáč and M. Bartušek (Inst. Anal. Chem., Univ. Brno, Czechoslovakia). *Z. anal. Chem.*, 1960, **178** (3), 198–201 (in German).—The reaction of Ni^{II} and Mn^{II} with formaldoxime (**I**) has been investigated; complexing occurs through the active polymeric form of **I**. The relative error of the method is <3%. *Photometric procedure*—To the neutral sample soln. (0·003 to 1 mg of Ni^{II} or Mn^{II}) add a 2% soln. of **I** (1 ml) and 1 ml of 10% NaOH soln. Dilute to 50 ml and measure the extinction at 470 mμ. *Polarographic procedure for Ni*—To the neutral sample soln. (0·006 to 1 mg of Ni) add 6 to 20 moles of **I** per g-atom of Ni, 1 ml of 0·1% gelatin soln. and 1 ml of N-KOH. Dilute to 10 ml, aerate with air or O, then displace this with N. Polarograph from −0·4 to −0·8 V *vs.* the S.C.E. Iron and Cu interfere. The reagent can be distilled directly from a mixture of hydroxylamine and excess of formaldehyde, or a soln. of the trimer (as hydrochloride) mixed with a soln. of $NaHCO_3$ of equal concn.
P. D. PARR-RICHARD

2884. **Oscillopolarographic behaviour of the platinum metals.** P. Beran (Dept. Anal. Chem., Charles' Univ., Prague). *Chem. Zvesti*, 1960 **14**, (11–12), 735–742.—Soln. of the platinum metals in various electrolytes were studied. With 2M-KBr or 2M-KI as electrolyte, Os (5×10^{-5}M) can be detected in the presence of Ru, the reaction of which is less sensitive (4×10^{-4}M). A mixture of Pd (0 to 1·2 mg), Rh (0·025 to 0·15 mg) and Ir (0·03 to 0·2 mg) in 10 ml can be analysed in M-1,2-diaminoethane sulphate by the use of comparative titrations with an error of ±10%; Pt^{II} and Pt^{IV} can be differentiated in this medium. In soln. of strong acids (*e.g.*, HCl), Ru, Rh, Pt and Ir produce a characteristic "thorn" on the curve $dE/dt = f(E)$, which is chiefly suitable for the sensitive detection of Rh (pD = 7·5). In a vol. of 20 ml containing 0·1M-HCl and 0·6M-NaCl, 0·5 to 25 μg of Rh can be determined in the presence of a 2000-fold excess of Pd or Pt and a 3300-fold excess of Au by the use of the method of standard addition. J. ZÝKA

2885. **Diallyldithiocarbamidohydrazine [*NN'*-di(allylthiocarbamoyl)hydrazine] (Dalzin) as an analytical reagent. Determination of palladium and its separation from nickel, zinc and copper.** N. K. Dutt and K. P. Sen Sarma (Univ. Coll. of Science, Calcutta, India). *Anal. Chim. Acta*, 1960, **23** (6), 531–533 (in English).—*NN'*-Di(allylthiocarbamoyl)-hydrazine (**I**) forms with Pd^{2+} a complex of the empirical composition $Pd(C_8H_{12}N_4S_2).H_2O$ which is pptd. quant. from aq. soln. at pH 3·1 to 4·5 *Procedure*—Neutralise the sample soln., containing 30 to 60 mg of Pd, with aq. NH_3, dilute to 100 ml and adjust the pH to ≃4 with citric acid. Heat on a water bath and add, with stirring, 25 ml of a hot 1% ethanolic soln. of **I**. Filter off the brick-red ppt. on a sintered glass crucible, wash with hot water, then with acetone, dry at 105°, and weigh. Other platinum metals, Be, Mg, Ca, Ba, Sr, Th, Ce, Mn, Ni and Zn remain in the filtrate, in which Ni and Zn can be determined as previously described (*Anal. Abstr.*, 1956, **3**, 3539). Copper, Hg and Ag interfere, but interference from Cu can be prevented by adding EDTA. H. N. S.

2886. **Investigations with iridium-192 of separations of platinum and rhodium from iridium. I.** K. W. Lloyd and D. F. C. Morris (Brunel College of Technology, London, W.3, England). *Talanta*, 1960, **7** (1–2), 117–120 (in English).—The degree of separation of Ir from Pt and Rh by the chromatographic procedure of Payne (*Analyst*, 1960, **85**, 698) is comparable with that by the method of Gilchrist and Wickers (*J. Amer. Chem. Soc.*, 1935, **57**, 2565). The former procedure is advocated in view of its greater speed. T. R. ANDREW

2887. **Methods of isotope analysis in geological samples for oxygen-18 and carbon-13.** R. Vedder (Inst. f. Physikalische Stofftrennung, Leipzig, Germany). *Kernenergie*, 1960, **3**, 890–892 (in German).—For the isotopic determination of C and O in carbonate rocks the CO_2 must be released from the carbonate. The relative abundances of ^{13}C and ^{18}O are measured by mass spectrometry. Various methods for the decomposition of carbonate rocks, problems of possible isotopic exchange reactions and isotopic fractionation processes are discussed. Acid and thermal decomposition are chiefly used. NUCL. SCI. ABSTR.

See also Abstracts—**2945**, Arsenic in coal and coke. **3046**, ^{90}Sr and ^{90}Y in natural water. **3047**, Ti in natural water. **3050**, Phosphate in hot springs. **3068**, Detection of F^- in gases. **3099**, Use of Tesla spark source in spectrography.

3.—ORGANIC ANALYSIS

Determination of elements and radicals and of organic compounds not included in other sections. Organic industrial products, including petroleum and its products, fuels, detergents, volatile oils, cosmetics, dyestuffs, fibres, plastics, resins, paints, elastomers, leather and explosives.

2888. **Indirect coulometric titration of reducible organic compounds by electrolytically generated ceric ions.** Takeo Takahashi and Hiroshi Sakurai (Inst. of Ind. Sci., Tokyo Univ., Yayoi-cho, Chiba). *J. Chem. Soc. Japan, Ind. Chem. Sect.*, 1960, **63** (4), 608–611.—The oxidation of Ce^{3+} and the reduction of Ce^{4+} proceed with the same current efficiency in the presence of $Fe_2(SO_4)_3$ (equiv. to the Ce) in N-H_2SO_4. Organic compounds that undergo a slow redox reaction with Ce^{4+} are caused to react with an excess of electrolytically generated Ce^{4+} under suitable conditions and the excess of Ce is determined coulometrically. 2-Naphthylamine, aniline and phenol are quant. oxidised with Ce^{4+} in N-H_2SO_4 at room temp., whereas oxalic acid, D-mannitol and D-sorbitol are oxidised at boiling-point.

Glutamic acid is not directly oxidised by Ce^{4+}, but a hydroxycarboxylic acid produced by the action of HNO_2 (30% $NaNO_2$, 2 ml) is oxidised at boiling-point in N-H_2SO_4. K. SAITO

2889. **Recommended test substances for the micro-determination of carbon and hydrogen.** International Union of Pure and Applied Chemistry. Commission on Microchemical Techniques. *Pure and Appl. Chem.*, 1960, **1** (1), 143–145.—The 38 compounds listed include those having extreme values for C and/or H, a variety of structural types and those incorporating elements that may interfere in the determination of C and H.

G. J. HUNTER

2890. **Rapid gas-chromatographic method for determination of carbon and hydrogen.** A. M. Vogel and J. J. Quattrone, jun. (Chem. Dept., Adelphi College, Garden City, N.Y.). *Anal. Chem.*, 1960, **32** (13), 1754–1757.—A rapid and simple method is described. The products of a bomb combustion are passed through a 2-metre gas-chromatographic column of dodecyl phthalate on diatomaceous earth and the percentages of C and H are calculated from the areas of the peaks obtained for H_2O and CO_2. Means and standard deviations for C determinations by this method are about half as good as those obtained by the Pregl method. For H determinations the accuracy is greater than that of the Pregl method.

C. B. BAINES

2891. **Organic micro-analysis. Studies on external absorbents of nitrogen oxides for the micro-determination of carbon and hydrogen. I.** Toshihiro Onoe (Takamine Lab., Sankyo Co., Ltd., Shinagawa, Tokyo). *Ann. Rep. Takamine Lab.*, 1958, **10**, 128–131.—The efficacy of external absorbents of nitrogen oxides produced during the combustion of N-containing organic compounds was examined by passing a current of nitrogen oxide through the absorbent and measuring the change in pH of an alkaline soln. in which the unabsorbed gas was trapped. Manganese dioxide, silica gel, PbO_2, hydroxyammonium chloride, $KMnO_4$, and a mixture of $K_3Fe(CN)_6$ and $K_4Fe(CN)_6$ were examined, the first two being the best.

II. Toshihiro Onoe. *Ibid.*, 1958, **10**, 132–135.—The absorptive capacity of MnO_2, which varies according to the conditions of preparation, was examined. Active MnO_2 prepared from $MnSO_4$ and $KMnO_4$ gave the best results.

III. Toshihiro Onoe. *Ibid.*, 1958, **10**, 136–142.—The absorption of nitrogen oxides with MnO_2 appeared to follow the equations $MnO_2 + 2NO = Mn(NO_2)_2$, and $MnO_2 + 2NO_2 = Mn(NO_3)_2$. The absorbent, before and after the reaction, was examined for the presence of NO_3^- and NO_2^- and by electron microscopy and X-ray diffraction. The X-ray diffraction pattern showed that the inner parts of the particles of MnO_2 had not been affected at a flow-rate of air in the combustion tube of 8 ml per min. Porous MnO_2 having a larger surface gave better absorption. S. NATORI

2892. **Method for the detection of oxygen in organic compounds.** B. Smith and R. Ohlson (Inst. f. Org. Kemi, Chalmers Tekn. Högskola, Göteborg, Sweden). *Acta Chem. Scand.*, 1960, **14** (10), 2245–2246 (in English).—The sample is decomposed by contact with a glowing platinum wire in a current of He or O-free N and the products of pyrolysis are passed through (*i*) a tube containing silica gel and (*ii*) a tube containing silica gel impregnated with a mixture of palladium sulphate and ammonium molybdate (*cf.* Shepherd, *Brit. Abstr. C*, 1948, 2). The yellow Pd - molybdosilicate complex is turned green or blue by the CO produced by the pyrolysis of O-containing organic compounds under the conditions described. Reducing substances other than CO, which might give a false positive result, are absorbed in the first silica-gel tube. The method is claimed to be specific, simple and reasonably reliable. I. A. SIMPSON

2893. **A simple, fast, and reliable Schütze - Unterzaucher method for the determination of oxygen in organic compounds.** W. J. Kirsten (Inst. of Med. Chem., Univ. of Uppsala, Sweden). *Microchem. J.*, 1960, **4** (4), 501–514.—A detailed description is given of the apparatus and its preparation. Interfering substances from compounds containing sulphur and halogens are removed by a liquid-oxygen trap. The CO formed is oxidised with anhydro-iodic acid—

$$15CO + 2(HIO_3.I_2O_5) \rightarrow 15CO_2 + 3I_2 + H_2O,$$

and the method is made simpler and more sensitive by absorbing and weighing all the reaction products in a CH-type absorption tube filled with Ascarite and Dehydrite. The results correspond to a gravimetric conversion factor of 0·1688.

A. J. BENNETT

2894. **Applications of infra-red spectroscopy. II. Observations on some aspects of the Zeisel alkoxyl determination.** D. M. W. Anderson and J. L. Duncan (Dept. of Chem., Univ., Edinburgh, Scotland). *Talanta*, 1960, **7** (1–2), 70–79 (in English).—The i.r. procedure of Anderson (*Analyst*, 1959, **85**, 50) has been applied to the study of the products present in the various stages of the Zeisel method under different experimental conditions. Water is inferior to soda asbestos as an absorbent of reaction products other than alkyl iodides (*e.g.*, HI, iodine, H_2S and CO_2). T. R. ANDREW

2895. **Application of enzymatic methods to organic functional-group analysis. I. Micro-determination of the primary amino-group.** T. S. Ma and R. Breyer (Dept. of Chem., Brooklyn College, New York, U.S.A.). *Microchem. J.*, 1960, **4** (4), 481–489.—In the presence of pyrophosphate buffered to pH 8·3 (8 ml of N-HCl and 100 ml of 2M-$Na_4P_2O_7$), D-amino-acid oxidase can deaminate a large number of D-α-amino-acids having a hydrogen atom and an amino-group on the α-carbon atom. The reaction is driven to completion by the use of excess of enzyme and sufficient catalase to destroy the H_2O_2 produced. *Procedure*—To the amino-acid (3 to 8 mg) in a micro-Kjeldahl flask add 10 times as much D-amino-acid oxidase, followed by pyrophosphate buffer (5 ml) and catalase (0·5 ml). Incubate at 35° to 37·5°, with occasional shaking. When the reaction is complete add 12 ml of a soln. containing 10 g of K_2CO_3 and 50 g of $Na_2B_4O_7$ per litre. Steam-distil the NH_3 into 2% H_3BO_3 soln. (5 ml), and titrate with 0·01N-HCl, with a methyl red - bromocresol green indicator. The K_2CO_3 - $Na_2B_4O_7$ soln. does not liberate NH_3 from amides and amino-acids that have not been attacked by the enzyme. The accuracy is ≃5%, and the presence of a large excess of the L-isomers does not cause interference.

A. J. BENNETT

2896. **Spectrophotometric methods for olefins. Colorimetric determination of conjugated diolefins.** A. P. Altshuller and I. R. Cohen (R. A. Taft Sanit. Engng Center, U.S. Dept. of Health, Education and Welfare, Cincinnati, Ohio). *Anal. Chem.*, 1960, **32** (13), 1843–1848.—Conjugated diolefins can be recovered from dilute mixtures with air and determined spectrophotometrically by coupling with *p*-nitrophenyldiazonium fluoroborate in a 2-methoxyethanol - H_3PO_4 solvent. With isoprene-type compounds the extinction at 490 mμ obeys Beer's law at concn. between 0·3 and 30 μg per ml. With buta-1,3-diene the absorption is linear with concn. between 20 and 200 μg per ml at 405 mμ. Aldehydes and ketones interfere with the determination of butadiene. Other hydrocarbons likely to be present do not interfere significantly.

G. BURGER

2897. **Spectrophotometric procedure for the microdetermination of methyl chloride.** M. Redford-Ellis and J. E. Kench (Dept. of Pathol., Manchester Univ., England). *Anal. Chem.*, 1960, **32** (13), 1803–1807.—The mechanism of a modified Fujiwara reaction (*S.B. Naturf. Ges. Rostock*, 1914, **6**, 33), in which a halogenated hydrocarbon is heated with pyridine and alkali to form a coloured compound, was studied. A procedure was developed for the determination of methyl chloride in the range 2 to 80 μg as a gas or in aq. soln. The reaction product absorbs strongly at 365 mμ and obeys Beer's law. The pyridine used must be purified by fractionation, oxidation and redistillation over P_2O_5. The reaction is carried out under specially purified O-free N, and is extremely sensitive to O. The procedure is precise but not specific for methyl chloride, interference from all other alkyl halides being likely.

G. BURGER

2898. **The application of gas chromatography to the determination of retention data and activity coefficients of some alkanes, alkyl nitrates, nitroalkanes and alcohols on selected stationary phases.** S. Evered and F. H. Pollard (Univ., Bristol, England). *J. Chromatography*, 1960, **4** (6), 451–457 (in English).—Data are presented for alkanes, nitroalkanes and alkyl nitrates on squalane and dinonyl phthalate (20% w/w on crushed firebrick) and for nitroalkanes and alcohols on diglycerol. The relative separation efficiencies and heats of solution for some nitroalkanes and alkyl nitrates in squalane and dinonyl phthalate are calculated.

W. T. CARTER

2899. **Identification of oxygen compounds in gas - liquid chromatographic fractions by catalytic deoxygenation.** C. J. Thompson, H. J. Coleman, R. L. Hopkins, C. C. Ward and H. T. Rall (Petroleum Res. Center, Bureau of Mines, U.S. Dept. of the Interior, Bartlesville, Okla.). *Anal. Chem.*, 1960, **32** (13), 1762–1765.—Oxygen compounds, such as alkyl and cyclic ethers, ketones, aldehydes and, in part, alcohols, are deoxygenated by a technique described earlier (*Anal. Abstr.*, 1960, **7**, 4811), and the resulting hydrocarbons are isolated and identified, thus leading to the identification of the oxygenated precursor. Fractions from a gas chromatograph are trapped as they emerge from the column, swept by a stream of H on to heated palladium and then transferred to the original gas-chromatographic column. Chromatograms obtained by the deoxygenation of pure and impure compounds are presented.

C. B. BAINES

2900. **Micro-analysis of volatile oils. V. Determination of methanol and ethanol in dilute solutions.** T. Beyrich and R. Pohloudek-Fabini (Inst. für Ernährung, Potsdam-Rehbrücke). *Ernährungsforschung*, 1960, **5** (4), 441–454.—Methods were developed for determining small amounts of methanol (up to 4 mg) and ethanol (up to 2 mg) in very dilute mixed soln. For methanol, up to 5 ml of soln. is transferred to a 10-ml tube and 2 ml of 25% ethanol is added, followed by H_2O to 10 ml. Of this, 2 ml is well shaken in a 20-ml stoppered tube with exactly 5 ml of $KMnO_4$ - H_3PO_4 soln. (3·16 g of $KMnO_4$ in 60 ml of H_2O diluted to 100 ml with M-H_3PO_4), and after 15 min. treated with 2 ml of oxalic acid - H_2SO_4 soln. (6·3 g of oxalic acid and 10 g of conc. H_2SO_4 in H_2O to 100 ml) and again shaken. The stopper is raised and left loose to allow escape of CO_2. After 15 min. the soln. is colourless (any slight ppt. is dissolved by closing the tube and tilting once or twice, the stopper being again loosened) and is made up to 10 ml and shaken. A 1-ml aliquot is added to a glass-stoppered test-tube, 1 ml of chromotropic acid soln. (1·5 g of sodium salt made up to 100 ml with H_2O) is added, and 8 ml of H_2SO_4 (1 vol. of H_2O with 3 vol. of conc. acid) is run in slowly from a burette. After mixing well, the soln. is heated for 15 min. in a water bath controlled at 60°, and the extinction of the cooled soln. is measured in a 10-mm cell at 570 mμ against a comparison soln. prepared from 2 ml of 25% ethanol diluted to 10 ml with H_2O. The calibration curve is linear up to 4 mg of methanol. For the determination of ethanol, up to 10 ml of soln. is diluted to 10 ml with H_2O and distilled with 1 ml of $K_2Cr_2O_7$ - H_2SO_4 soln. (60 g of $K_2Cr_2O_7$, 50 g of conc. H_2SO_4 and 600 g of H_2O) and 5 ml of 4N-H_2SO_4 in a stream of N into two closed receivers (each containing 1 ml of H_2O cooled in ice) at a rate of 5 ml per 15 min. The contents of the receivers are combined and made up to 10 ml, 0·5 ml of sodium nitroprusside soln. (4%) and 1·5 ml of piperazine hydrate soln. (25%) are added, the mixture is well shaken for 30 sec. and the extinction at 560 mμ is measured after 3 min. or at 30-sec. intervals until the max. is reached. Results accurate to within ±15% were obtained on test mixtures. (31 references.)

C. L. HINTON

2901. **Colorimetric semi-micro determination of isopropyl alcohol.** G. B. Ginther and R. C. Finch (Eaton Lab., Norwich Pharmacal Co., Norwich, N.Y.). *Anal. Chem.*, 1960, **32** (13), 1894–1895.—A procedure is given for the determination of isopropyl alcohol in aq. soln. To 5 ml of the test soln. (0·1 to 1·0 mg of isopropyl alcohol) add 1 ml of 1% $K_2S_2O_8$ soln. and heat in a closed flask for 15 min. at 80°. Cool, add 1 ml of 5% $NaHSO_3$ soln. and mix. Add 4 ml of 40% NaOH soln. and 1 ml of 20% ethanolic salicylaldehyde soln. and shake. Heat the soln. for 15 min. at 80°, cool and dilute to 25 ml. Measure the extinction against a blank at any wavelength between 470 and 530 mμ and compare with a standard curve. The blank is yellow; isopropyl alcohol gives an orange product. Compounds that yield acetone by oxidation interfere. Some interference is caused by isobutyl and t-butyl alcohols. Methanol, ethanol and n-propanol do not interfere.

G. BURGER

2902. **Infra-red spectroscopic detection of small amounts of water in aliphatic alcohols and ketones.** E. Wiegel and H. H. Kirchner (Physik.-Techn. Bundesanstalt, Braunschweig, Germany). *Z. anal.*

Chem., 1961, **178** (4), 241–260 (in German).—Some results are reported of a systematic investigation of the determination of small amounts of H_2O in methanol, ethanol, n-propanol, isopropyl alcohol, n-butanol, isobutyl alcohol, acetone and butanone, by i.r. spectroscopy. The OH-deformation, OH-valence and OH-combination vibrations of H_2O in aliphatic solvents can be used for the detection and determination of H_2O (0·1 to 10%, v/v). For H_2O concn. <5%, cells with sodium chloride windows and, for concn. >5%, cells with arsenic trisulphide windows, are used. B. B. BAUMINGER

2903. **Determination of ethylene and propylene glycols in mixtures by gas chromatography.** H. G. Nadeau and D. M. Oaks (Olin Mathieson Chem. Corp., New Haven, Conn.). *Anal. Chem.*, 1960, **32** (13), 1760–1762.—A rapid, accurate and convenient method is described that shows no interference from water, diethylene glycol, triethylene glycol, dipropylene glycol or hexane-1,2,6-triol. Separation is achieved on a 2-metre column of 5% tetrahydroxyethylenediamine on Chromosorb W at 105° with a thermistor-type detector. The method can be extended to the determination of the two glycols in soln. containing 90% of water. Calibration data and typical chromatograms are presented. C. B. BAINES

2904. **Colorimetry of sugars by the camphor-sulphuric acid reaction.** Y. Matsuo and A. Nanba. *J. Ferment. Technol., Japan*, 1960, **38**, 188–192.—The colorimetric method of Hadi for determining sucrose by reaction with a 1% soln. of camphor in H_2SO_4 (*Anal. Abstr.*, 1959, **6**, 2204) was tested on fructose, glucose and xylose soln. In each case, 2 ml of the soln. in a cooled test-tube was slowly mixed with 3 ml of reagent. The tube was then heated in a constant-temp. bath and cooled; the extinction was then measured within 10 min. in a photo-electric colorimeter. With glucose and a reagent prepared with conc. H_2SO_4, heating for 20 min. at 100° was needed, but the blank test then also gave a strong colour, so that the method is unsuitable for glucose. With a reagent prepared with H_2SO_4 (3:1), fructose and xylose could be determined in the presence of glucose. Fructose soln. required heating at 60° for 30 min., the colour being measured at 500 mμ; 0·005% could be determined in the presence of 10 times as much glucose with an error of ≯4%. Xylose (0·005%) could be determined with the same accuracy in the presence of up to five times as much glucose, the colour being measured at 470 mμ after heating at 60° for 3 hr. For fructose or xylose soln. in the absence of glucose, heating with the modified reagent at 100° for 6 min. and 10 min., respectively, was found to be suitable. Fructose could be determined in the presence of 50 times as much xylose by heating for 30 min. at 60°.

SUGAR IND. ABSTR.

2905. **Chromatographic analysis of sugars as complex borate ions.** A. Hallén (Chem. Dept. II, Karolinska Inst., Stockholm, Sweden). *Acta Chem. Scand.*, 1960, **14** (10), 2249–2250 (in English).—Neutral sugars can be separated chromatographically as borate complexes on an anion-exchange resin and eluted with dilute borate buffer. The resin, Dowex 2-X8 (200 to 400 mesh), after treatment successively with 2M-HCl, 2M-NaOH, 2M-Na_2CO_3 and finally with 0·03M-$Na_2B_4O_7$ in 0·6M-$NaHCO_3$, is suspended in a soln. of 0·1M-$Na_2B_4O_7$ in 0·2M-$NaHCO_3$ and allowed to sediment spontaneously to form the chromatographic column (6 mm × 150 cm). The mixture of sugars (0·2 to 0·8 mg of each) is dissolved in 0·05M-$Na_2B_4O_7$ (0·3 ml) and the soln. is passed through the column at a flow rate of 1·5 ml per hr. The neutral sugars are eluted at room temp. with a soln. of 0·01M-$Na_2B_4O_7$ in 0·2M-$NaHCO_3$ and the uronic sugars are then eluted with 0·03M-$Na_2B_4O_7$ in 0·6M-$NaHCO_3$. Both soln. have a pH of 8·7. To determine the sugars, each eluate fraction (1 ml) is heated for 20 min. in a boiling-water bath with 3 ml of a fresh mixture of aniline (10 ml) in acetic acid (500 ml). The intensity of the yellow colour is compared at 370 mμ with that developed by a standard sugar soln. A set of standards is prepared for each sugar, containing 10 to 100 μg of sugar per ml, dissolved in a soln. of 0·01M-$Na_2B_4O_7$ in 0·2M-$NaHCO_3$. I. A. SIMPSON

2906. **Determination of pentosans. VI. Differential u.v. spectrophotometric method for the determination of furfuraldehyde and 5-hydroxymethylfurfuraldehyde in Tollens distillates.** P. O. Bethge (Swedish Forest Products Res. Lab., Stockholm, Sweden). *Svensk Papp-Tidn.*, 1960, **63** (22), 813–816 (in English).—A two-component differential spectrophotometric method is described that permits the analysis of mixtures of two components having similar spectra, which cannot be analysed by conventional two-component spectrophotometry. The method has been applied to the determination of furfuraldehyde in Tollens distillates, and the accuracy and precision are calculated. Simplified procedures applicable to long series of similar samples are discussed. P. PEDERSEN

2907. **Problems of the paper chromatography of starch.** M. Ulmann and M. Richter (Inst. für Ernährung, Potsdam-Rehbrücke). *Ernährungsforschung*, 1960, **5** (4), 462–477.—The nature of the heterogeneity of starch is discussed, and an account is given of methods used hitherto in attempts to separate and characterise homogeneous individual constituents. By a suitable technique of ascending paper chromatography applied to thermal or alkali dispersions of potato starch, with 0·2N-NaOH as the most useful of a number of mobile phases, a fractionation of the amylose into two, and of the amylopectin into two, and under some conditions three, fractions was observable. Further work is required to establish whether the fractionation is due to constitutional or polymerisational differences, but indications were obtained that mol. wt. was not decisive as regards mobility. The possibility of a special effect of the alkali during the dispersion of the starch in exposing a weakly branched amylopectin is discussed. (28 references.) C. L. HINTON

2908. **Determination of formaldehyde by gas chromatography.** S. Sandler and R. Strom (Dept. of Chem. and Mech. Engng, Univ., Toronto, Ont., Canada). *Anal. Chem.*, 1960, **32** (13), 1890–1891.—Two columns are described with surfactants extracted from commercial detergents as the stationary phase. A column suitable for the separation and determination of formaldehyde from its soln. in water consisted of a mixture of 30 to 60-mesh Fluoropak 80 coated with 10% (w/w) of Tide surfactant and 30 to 60-mesh sand coated with 1% (w/w) of the same surfactant (2:1). A second column suitable for the qual. analysis of complex soln. consisted of 30 to 60-mesh C22 firebrick coated with 30% (w/w) of Sail surfactant. C. B. BAINES

2909. **Determination of paraldehyde in aqueous solutions of acetaldehyde.** H. Pobiner (Esso Res. and Engng Co., Linden, N.J., U.S.A.). *Anal. Chem.*, 1960, **32** (13), 1899.—Infra-red data are given.

2910. **The use of bromine chloride in analytical chemistry. Determination of unsaturated aldehydes.** K. Burger and E. Schulek (Inst. of Inorg. and Anal. Chem., L. Eötvös Univ., Budapest). *Talanta*, 1960, **7** (1–2), 46–50 (in English).—A standard soln. of BrCl may be used to react with the double bonds in unsaturated aldehydes without attacking the CHO group. Results are reported for cinnamaldehyde, crotonaldehyde and acraldehyde.

T. R. ANDREW

2911. **Spectrophotometric determination of malonaldehyde in aqueous solution.** Fujio Mashio and Yoshiko Kimura (Kyoto Tech. College, Matsugasaki, Sakyo-ku). *J. Chem. Soc. Japan, Pure Chem. Sect.*, 1960, **81** (3), 434–437.—Malonaldehyde (**I**) has a marked absorption at 245 mμ (molar extinction coeff. $1{\cdot}296 \times 10^4$) in acid soln. and at 267 mμ ($3{\cdot}003 \times 10^4$) in basic soln. Since this spectrum differs appreciably from that of acetaldehyde, it would appear that **I** is present in the tautomeric form β-hydroxyacraldehyde. The change in spectrum at pH 3 to 7 is due to the dissociation of the alcoholic hydroxyl group. The spectrophotometric analysis is carried out at pH <3 or >7.

K. SAITO

2912. **Fluorescence spot-tests for glyoxal, pyruvaldehyde, salicylaldehyde and some other aromatic aldehydes.** E. Sawicki and T. W. Stanley (Robert A. Taft Sanitary Engng Center, U.S. Dept. of Health, Educ. and Welfare, Cincinnati, Ohio). *Chemist Analyst*, 1960, **49** (4), 107 (in English).—The hydrazones of salicylaldehyde and 2-hydroxynaphthaldehyde (preparation described) form fluorescent azines with glyoxal, pyruvaldehyde and some aromatic aldehydes. The azine is prepared by spotting the test soln. on to filter-paper impregnated with the reagent, and is detected with u.v. light. Many polynuclear aromatic aldehydes can be detected by treating the paper with conc. HCl after the addition of the test soln. The limits of detection vary from 0·01 to 10 μg, depending on the aldehyde.

W. T. CARTER

2913. **Non-aqueous acidification in preparation of organic acid concentrates for gas chromatography.** I. R. Hunter, H. Ng and J. W. Pence (Western Regional Res. Lab., Albany, Calif.). *Anal. Chem.*, 1960, **32** (13), 1757–1759.—A method for the detection and identification of small amounts of volatile food flavours is based on the preparation of concentrated anhydrous extracts of the carboxylic acids present therein. Concentrates containing sodium salts of organic acids are acidified with dichloroacetic acid in acetone to liberate the free acids, which dissolve in the acetone. The free acids are then separated on a column of 5% of LAC IR 296 (glycol - adipate polymer) on firebrick. The method is applied to the extraction and preparation for analysis of the acids produced in a bread preferment liquid; 12 acids are identified.

C. B. BAINES

2914. **Gas-chromatographic characterisation of fatty acids. Identification constants for mono- and di-carboxylic methyl esters.** T. K. Miwa, K. L. Mikolajczak, F. R. Earle and I. A. Wolff (Agric. Res. Service, U.S. Dept. of Agric., 1815 North University, Peoria, Ill.). *Anal. Chem.*, 1960, **32** (13), 1739–1742.—A technique is described whereby each component on the chromatogram is expressed as a readily reproducible constant ("equivalent chain-length"), which is independent of experimental conditions for a given stationary phase and carrier gas. A combination of two values obtained by use of a polar column (Resoflex 446 on Celite) and a non-polar column (Apiezon L on Celite) is sufficient to characterise most fatty acids. The utility and applicability of the method to a wide variety of lipid materials, and to the consistent changes in parameters ascribable to specific functional groups, are discussed. Equivalent chain-length values are given for a number of monocarboxylic methyl esters for use as standards, and of the monocarboxylic methyl esters of fatty acids occurring in seed oils.

C. B. BAINES

2915. **Low-temperature oxidation of oxalic acid with potassium permanganate.** I. M. Issa, A. A. El-Aasser and M. M. El-Merzibani (Chem. Dept., Univ. Assiut, Egypt). *Z. anal. Chem.*, 1960, **178** (1), 12–19 (in English).—It is shown that, in the presence of Mn^{2+} and Cu^{2+}, oxalic acid can be titrated with $KMnO_4$ in the cold; the end-point is the appearance of a lilac or faint-violet coloration. Other catalysts, *e.g.*, Zn^{2+}, Cd^{2+}, Hg^{2+} and Ag^+, are less efficient. The effects of $CuSO_4$ concn., acidity, Mn concn., temp. and oxalic acid concn. have been studied. The results are tabulated.

B. B. BAUMINGER

2916. **Determination of fermentation citric acid.** J. Bártfai (Res. Inst. for the Fermentation Ind., Budapest). *Magyar Kém. Foly.*, 1960, **15** (12), 566–567.—A modification of the method of Pucker *et al.* (*J. Biol. Chem.*, 1936, **113**, 235) is described, in which the pentabromoacetone (**I**) formed by oxidation of the citric acid in the presence of Br is extracted with $CHCl_3$. After evaporation of the $CHCl_3$, the residual **I** is weighed. Comparative tests show the superiority of $CHCl_3$ to light petroleum used as extraction medium in the original method, the results being more reproducible. When known amounts of pure citric acid were determined by the modified procedure, the yield of **I** was 95 to 96%, as against 90% when light petroleum was used.

B. VASZKO

2917. **Determination of the purity of malonic ester by gas chromatography.** K. Dorfner (Ammoniaklab., Badischen Anilin- u. Soda-Fabrik, Ludwigshafen-am-Rhein, Germany). *J. Chromatography*, 1960, **4** (6), 502–503 (in German).—Esters of malonic, methylmalonic and dimethylmalonic acids can be separated on a 10-metre column of 24% silicone grease on Celite 535 at 180°. No separation is obtained on a similar column packed with Chromosorb as the inert support.

W. T. CARTER

2918. **Determination of thiourea.** J. F. Alicino (Squibb Inst. Med. Res., New Brunswick, N.J., U.S.A.). *Microchem. J.*, 1960, **4** (4), 551–553.—Dissolve the sample (3 to 5 mg) in glacial acetic acid (5 ml) and add 1 ml of mercuric acetate reagent (30 g of mercuric acetate in 1 litre of glacial acetic acid). Add 1 drop of indicator and titrate with 0·01N-$HClO_4$ in glacial acetic acid. Crystal violet, 0·1% in glacial acetic acid, may be used as indicator, but 0·1% quinaldine red in glacial acetic acid gives a sharper end-point (1 ml of 0·01N-$HClO_4 \equiv 0{\cdot}761$ mg of thiourea). The blank titration should be $<0{\cdot}1$ ml of 0·01N-$HClO_4$.

A. J. BENNETT

2919. **Polarographic determination of thioethanolamine [2-aminoethanethiol] and its disulphide in the presence of one another.** E. C. Chevalier and W. C. Purdy (Maryland Univ., College Park, U.S.A.).—In a Clark and Lubs buffer soln. of pH 7·4, 2-aminoethanethiol (**I**) has a $E_{\frac{1}{2}}$ value of −0·420 V *vs.* the S.C.E. and its disulphide (**II**) a $E_{\frac{1}{2}}$ value of −0·620 V. **I** gives a linear current - concn. relationship over the range 2×10^{-5} to 10^{-3}M and **II** over the range $7{\cdot}5 \times 10^{-6}$ to $7{\cdot}5 \times 10^{-3}$M. **I** can be determined in the presence of a 10-fold excess of **II**, and **II** in the presence of a 100-fold excess of **I**.
H. N. S.

2920. **Analysis of some volatile phosphorus compounds by gas - liquid chromatography.** S. H. Shipotofsky (Kansas State Univ., Manhattan). *U.S. Atomic Energy Comm.*, Rep. TID-6437, 1960. 22 pp.—A gas-chromatographic method for the analysis of mixtures of dimethyl and diethyl phosphite, PCl_3 and $PSCl_3$, or PCl_3 and $POCl_3$ was developed. Pyrex glass was used for the column and for the body of the thermal-conductivity cell, and tantalum wire was employed for the sensing elements.
NUCL. SCI. ABSTR.

2921. **Quantitative determination of phosphonium salts and phosphoranes.** S. T. Ross and D. B. Denney (State Univ., Rutgers, New Brunswick, N.J.). *Anal. Chem.*, 1960, **32** (13), 1896.—Satisfactory results are reported for the determination of phosphonium salts and phosphoranes by titration in acetic acid with 0·02N-$HClO_4$ in acetic acid, with crystal violet as indicator, by the technique of Fritz and Hammond ("Quantitative Organic Analysis," Wiley, N.Y., 1957, Chap. 3). For the determination of phosphonium salts, Hg acetate is added to the acetic acid soln., and the liberated acetate ion is titrated.
G. BURGER

2922. **Improvement in the determination of a trace of thiophen in benzene by the indophenin colour reaction.** Eizo Yasui and Hiroshi Suzuki (Devel. Dept., Toa Gosei Chem. Ind., Showa-cho, Minato-ku, Nagoya). *J. Chem. Soc. Japan, Ind. Chem. Sect.*, 1960, **63** (4), 612–615.—The extinction of isatin (**I**) in conc. H_2SO_4 (10 mg per 100 ml) containing Fe^{3+} (25 mg) changes with time and becomes constant after 8 days. When this soln. is treated with a sample containing thiophen, the extinction is greatly reduced; the sample and reagent must be shaken immediately after mixing; $>2{\cdot}5$ μg of NO_2^- per ml of H_2SO_4 interferes. The error is <10% and the result is not affected by temp. *Procedure*—Shake benzene (1 ml, containing 0·00001 to 0·01% of thiophen) with **I** soln. in H_2SO_4 [700 ml of conc. H_2SO_4 containing 0·1 g of **I** mixed with 34 ml of 6N-H_2SO_4 containing 0·25 g of $(NH_4)_2SO_4.Fe_2(SO_4)_3$ and 266 ml of conc. H_2SO_4 and set aside for 8 days] (10 ml) on a shaker (110 vibrations per min., amplitude 50 mm) for 10 min. and measure the extinction of the acid layer at 540 mμ after 2 hr. against the reagent soln.
K. SAITO

2923. **Analysis of aromatic hydrocarbons by means of paper chromatography. I. Isolation and identification of n-alkyl-substituted benzenes in the form of sulphonic acids.** D. Vulchev and D. Elenkov (Chem.-tech. Inst., Sofia, Bulgaria). *Compt. Rend. Acad. Bulg. Sci.*, 1960, **13** (6), 677–679 (in English).—The n-alkylbenzenes (10 drops) having alkyl groups containing up to 5 C atoms are sulphonated with H_2SO_4 (10 drops) containing 5 to 8% of SO_3, and the homogeneous mixture is then diluted with aq. NaCl and chromatographed. Ascending chromatography on Whatman WF 1 paper with 1% aq. NH_3 satd. with butanol separates the constituents within 18 hr. The spots are detected by heating the chromatograms at 205° to 215° for 10 to 15 min. Toluene is used as internal standard for the determination of R_F values. Only the separation of butyl- from amyl-benzene presents difficulty.
J. P. STERN

2924. **Correlation of mass spectra with structure in aromatic oxygenated compounds. Aromatic alcohols and phenols.** T. Aczel and H. E. Lumpkin (Res. and Dev. Div., Humble Oil & Refining Co., Baytown, Tex., U.S.A.). *Anal. Chem.*, 1960, **32** (13), 1819–1822.—A systematic study is made of the correlations existing between the mass spectra and the chemical structure of 8 benzyl alcohols, 11 monohydroxybenzenes and 3 dihydroxybenzenes. Spectral features are correlated with the stability and fragmentation pattern of the benzene ring and the nature, number and position of the alkyl substituents. Qual. and quant. analysis of mixtures, and the possibility of prediction of the sensitivities of compounds at present unavailable for calibration are outlined. The influence of the position of the substituent groups can be used to distinguish isomeric compounds.
C. B. BAINES

2925. **Photometric titration of weak acids in non-aqueous media. Determination of phenols in isopropyl alcohol.** L. E. I. Hummelstedt and D. N. Hume (Dept. of Chem., Massachusetts Inst. of Technol., Cambridge 39, U.S.A.). *Anal. Chem.*, 1960, **32** (13), 1792–1797.—Phenols, approx. 0·01M in isopropyl alcohol, can be titrated photometrically with tetra-n-butylammonium hydroxide (**I**) in isopropyl alcohol. The titrant was prepared by evaporating most of the methanol from M-methanolic **I** and diluting the concentrate with isopropyl alcohol to give a 0·4M soln. containing 10 to 15% of methanol. The reagent must be protected from CO_2, and, for the titration of stronger acids, should be passed through an ion-exchange column (OH^- form). A list of appropriate wavelengths is given for 16 phenols. Up to 4 components of a mixture can be resolved in favourable cases by making wavelength shifts after each break in the curve of extinction *vs.* vol. of titrant. The resolution is more sensitive than with potentiometric or polarographic procedures. A combination of photometric and potentiometric titration is advantageous in some cases. The shape of the curves indicates complex formation.
G. BURGER

2926. **Gas-chromatographic analysis of chloromethylated phenyl ether.** W. B. Trapp, J. L. Pillepich and E. D. Ruby (The Dow Chemical Co., Midland, Mich.). *Anal. Chem.*, 1960, **32** (13), 1737–1739.—Thermally unstable chloromethylated phenyl ether is analysed by hydrogenolysis to the methyl-substituted phenyl ether, followed by gas chromatography on a 15-ft. column of 20% bis[α-(*x*-tolyl)-*p*-tolyl] ether on 30 to 60-mesh Chromosorb, with a thermal-conductivity detector. Eight components of the reaction mixture from the chloromethylation of phenyl ether have been quant. determined and an application of the method is described wherein the six major components are traced through the chloromethylation process to provide data for a kinetic profile of the reaction.
C. B. BAINES

2927. **Spot-test detection of phenoxy-compounds.** F. Feigl, V. Gentil and D. Haguenauer-Castro (Lab. da Prod. Mineral, Min. da Agric., Rio de Janeiro, Brazil). *Microchem. J.*, 1960, **4** (4), 445–450.—Add 10 mg of sodium azide to 1 or 2 drops of a fresh soln. of 10 mg of *NN*-diphenylbenzidine in 100 ml of conc. H_2SO_4. Add 1 drop of a soln. of the phenoxy-compound and mix. A blue colour is produced. The reaction is given by phenol (0·25 μg) and also by aliphatic and aromatic phenyl ethers and phenyl esters of carboxylic acids. Ether should not be used as a solvent because the peroxide frequently present interferes. The reaction is due to the intermediate formation of nitrosobenzene, which oxidises the reagent to a blue *p*-quinonoid compound. A. J. BENNETT

2928. **Coulometric titration of quinol and *p*-aminophenol by electrolytically generated ceric ions.** Takeo Takahashi and Hiroshi Sakurai (Inst. of Ind. Sci., Tokyo Univ., Yayoi-cho, Chiba). *J. Chem. Soc. Japan, Ind. Chem. Sect.*, 1960, **63** (4), 605–607.—Quinol is rapidly oxidised by Ce^{4+}, and its coulometric titration at 10 mA is carried out in N-H_2SO_4 containing 0·005N-$Ce_2(SO_4)_3$, the constant-current device of Carson (*Anal. Chem.*, 1950, **22**, 1565) being used as the current source. The time required for the electrolysis decreases with increase in concn. of H_2SO_4 and $Ce_2(SO_4)_3$, remaining constant at concn. >0·5M and >0·004N, respectively. Decrease in the rate of stirring increases the fluctuation. The current efficiency is 90 to 95%. *p*-Aminophenol is similarly titrated at 5 mA in 2N-H_2SO_4 containing 0·015N-$Ce_2(SO_4)_3$. K. SAITO

2929. **Mass spectra of various quinones and polycyclic ketones.** J. H. Beynon and A. E. Williams (I.C.I. Ltd., Dyestuffs Div., Manchester, England). *Appl. Spectroscopy*, 1960, **14** (6), 156–160.—The mass spectra of 21 substituted anthraquinones, 3 other quinones and 2 polycyclic ketones are given. The spectra are discussed from the point of view of understanding the processes involved in the fragmentation of compounds containing keto-groups and of distinguishing between various isomers and identifying unknown compounds in these classes. E. G. CUMMINS

2930. **Determination of hydroxybenzoic acids in potassium bromide discs.** D. E. Nicholson (Humble Oil & Refining Co., Baytown, Tex., U.S.A.). *Anal. Chem.*, 1960, **32** (13), 1899.—Infra-red data for the three isomers are given.

2931. **Effect of substituent groupings on the chromatographic behaviour of phenoxyacetic acids. II. Chloro-substituted phenoxyacetic acids.** L. S. Bark and R. J. T. Graham (Dept. of Chem. and Appl. Chem., Royal Tech. Coll., Salford, England). *Analyst*, 1960, **85**, 905–907.—Previous work with methyl-substituted phenoxyacetic acids (*Ibid.*, 1960, **85**, 663) has been extended to 12 chloro-substituted phenoxyacetic acids. Comparison of R_F values of the chloro- and methyl-substituted compounds suggests that substitution of a Cl atom *meta* to the phenoxyacetic group has a special effect on the mode of adsorption on the paper. Comparison of the results obtained for chloro-, methyl- and methoxy-substituted acids suggests that the acid may be adsorbed by attachment of the acid group and the substituent group, the substituent group being attached by hydrogen bonding between the hydroxyl groups of the cellulose and the electronegative atom of the substituent. The acids substituted in the 2- and 6-positions show relatively low adsorption, probably owing to reduction of rotation of the phenoxyacetic acid group and consequent attachment of this group only.

III. Effect of the nature of the substituent. L. S. Bark and R. J. T. Graham. *Ibid.*, 1960, **85**, 907–908.—To examine the effects of different substituents on the adsorption of the acids, a series of mono-substituted phenoxyacetic acids was chromatographed under standard conditions, the substituents being chosen so that both effect of size and electronegativity of the substituent group could be considered. The results indicated that the greater the electron density in the 4-position the greater is the effect of the substituent. The nature of the substituent group is of importance in determining the chromatographic behaviour of the molecule; the greater the probability of hydrogen-bond formation between the substituent and the stationary phase the greater will be the attachment to this phase and the lower the R_F value. A. O. JONES

2932. **Detection and determination of polynitro aromatic compounds.** E. Sawicki and T. W. Stanley (R. A. Taft San. Engng Center, U.S. Dept. of Health, Educ., and Welfare, Cincinnati, Ohio, U.S.A.). *Anal. Chim. Acta*, 1960, **23** (6), 551–556 (in English).—*Spot-test procedure*—To 1 drop of 25% aq. tetraethylammonium hydroxide (**I**) on a spot plate add 1 drop of a 5% soln. of fluorene in dimethylformamide (**II**) and 1 drop of a soln. of the test substance in **II**. A 50% soln. of butanone in **II** may be substituted for the fluorene soln. The reaction can also be carried out on paper. Many aromatic polynitro-compounds yield blue, green, violet, red or yellow colours. Such colours are also given by certain compounds that contain only one nitro-group or none at all, but which contain strongly electronegative groups. *Photometric procedure*—To 5 ml of the test soln. in **II** add 5 ml of 0·1% fluorene soln. in **II** and 1 ml of 10% aq. **I** and dilute to 50 ml with **II**. Determine the extinction at the appropriate wavelength within 3 min. Absorption maxima and mol. extinction coeff. are given for 5 dinitro-compounds. Some polynitro-compounds yield colours that are too unstable for the quant. procedure. H. N. S.

2933. **Determination of *N*-ethyl-*m*-toluidine in *N*-ethyl-*N*-(2,3-dihydroxypropyl)-*m*-toluidine.** K. B. Whetsel, W. E. Roberson and M. W. Krell (Tennessee Eastman Co., Kingsport, Tenn., U.S.A.). *Anal. Chem.*, 1960, **32** (13), 1899.—Infra-red data are given.

2934. **New analytical methods for the analysis of technical dibenzyl disulphide.** B. Klouček, V. Jehlička and J. Gasparič (Res. Inst. Org. Synth., Pardubice-Rybitví, Czechoslovakia). *Chem. Průmysl*, 1960, **10** (12), 624–626.—Methods for the determination of dibenzyl sulphide and dibenzyl tetrasulphide impurities and a volumetric determination of dibenzyl disulphide were developed. *Procedures*: (i) *Determination of dibenzyl disulphide*—Heat the sample (0·1 to 0·2 g) in 90% ethanol (25 ml) containing 0·15% of HCl at 60°. To the warm soln. add amalgamated zinc washed with H_2O and ethanol (mix 100 g of zinc with 45 ml of H_2O and 55 ml of conc. HCl, add 27 mg of $HgCl_2$ and shake for 1 min.; wash with H_2O) (10 to 20 ml) and heat to 65° to 67°. Add 6N-H_2SO_4 (4 ml) and set aside for 20 min. at 65°, with occasional shaking. Cool, separate the soln., wash the amalgam layer with

portions of a mixture of ethanol and H_2SO_4 (100 ml) (filter the last portions through paper) and titrate with 0·1N-iodine till the soln. becomes yellow - green. (*ii*) *Paper chromatography*—Place the sample soln. (2 g in 10 ml of ethanol) on WF 1 paper impregnated with 10% 1-bromonaphthalene soln. in ethanol and develop by the descending technique with 80% acetic acid satd. with 1-bromonaphthalene. Locate the spots with a soln. of K_2PtI_6 (0·5 ml of 5% $PtCl_4$ in N-HCl mixed with 45 ml of 10% KI soln. and 5 ml of H_2O) (pale-yellow spots of dibenzyl sulphide and yellow or yellow - brown spots of disulphide and polysulphides on a pink background). (*iii*) *Polarography of dibenzyl tetrasulphide*—Dissolve the sample (0·5 g) in benzene - methanol (1 : 1) and dilute with H_2O to 50 ml; mix an aliquot (1 to 10 ml) with 2M-acetate soln. (13·6 g of cryst. Na acetate plus 5·8 ml of anhyd. acetic acid dissolved in 250 ml of methanol and diluted to 500 ml with benzene) (10 ml) and record the polarographic wave from −0·2 to −1·2 V *vs.* the mercury pool. J. ZÝKA

2935. **Characterisation and determination of benzo[*a*]pyrene by luminescence spectrophotometry at −190°.** B. Muel and G. Lacroix (Lab. Pasteur de l'Inst. du Radium, rue d'Ulm, Paris). *Bull. Soc. Chim. France*, 1960, (11–12), 2139–2147.—The fluorescence spectrum of benzo[*a*]pyrene (**I**) in octane at −190° is shown to have much fine structure and to be more definitive than that obtained at normal temp. A qualitative comparison of spectra demonstrates that the material extracted from cigarette-smoke is **I**. Quantitative analyses are effected either by comparison with standard soln. or, better, by incremental additions of **I** to the sample soln. The accuracy is better than 10%. The methods have been applied to cigarette-smoke, distilled spirits (≃0·4 mμg per ml) and tap water (<0·003 mμg per ml). J. R. HUDSON

2936. **Detection of o-quinones and guanidine carbonate by pyro-synthesis in spot analysis.** F. Feigl and D. Goldstein (Lab. Prod. Mineral, Min. da Agric., Rio de Janeiro, Brazil). *Z. anal. Chem.*, 1961, **178** (4), 265–270 (in German).—The micro-detection of *o*-quinones (**I**) is effected by mixing in a micro test-tube a drop of the sample soln. in ethanol, ether, benzene or glycerol with a few mg of guanidine carbonate, warming to remove the solvent, and heating the tube in a glycerol bath at 180°. The condensation products formed are soluble in ethanol and are coloured; phenanthrenequinone (≮1·5 μg) gives a blue colour, 1,2-naphthaquinone (≮10 μg) a black colour and 4,7-phenanthroline-5,6-quinone (≮5 μg) a red colour. The procedure for the detection of guanidine carbonate (**II**) is similar to that for **I**, except that a drop of the sample in aq. soln. is treated with a drop of 0·1% phenanthraquinone soln. in benzene; 5 μg of **II** gives a positive response. When guanylguanidine (**III**) or dicyandiamide (**IV**) is heated with Na_2CO_3, guanidine is formed and is detected as described above; 12·5 μg of **III** and 25 μg of **IV** can be detected. The mechanism of the reactions is discussed.
B. B. BAUMINGER

2937. **Gas chromatography of pyridine bases: elimination of peak tailing.** W. E. Golding and C. A. Townsend (N. Thames Gas Board, Beckton, London, England). *Chem. & Ind.*, 1960, (48), 1476.—The peak-tailing which occurs in the analysis of coal-tar bases has been eliminated. The column support (Embacel) has been pre-treated by boiling under reflux with HCl for 3 hr., washing till free from acid and soaking in 5% Na_2CO_3 soln., then draining and drying at 110°. This coated support eliminates tailing, but satisfactory separation is achieved only when it is used in conjunction with a stationary phase consisting of polyoxyethylene glycol 400 and dinonyl phthalate (4 : 1, w/w). These materials, with an inlet pressure of 19 p.s.i.g. and a carrier-gas flow rate of ≃90 ml per min., enabled a good separation of bases boiling up to 172° to be achieved. E. G. CUMMINS

2938. **Ultra-violet spectrophotometric determination of quinoline and isoquinoline by a rapid compensation method.** Motohisa Furusawa and Tsugio Takeuchi (Inst. Tech.-anal. Chem., Fac. Engng, Nagoya Univ., Chikusa-ku, Nagoya). *J. Chem. Soc. Japan, Ind. Chem. Sect.*, 1960, **63** (4), 616–619.—The u.v. spectrophotometric analysis of binary mixtures of quinoline (**I**) and isoquinoline (**II**), in which one component predominates, is improved by the use of a soln. of the predominating component as reference. The extinctions of **I** or **II** (<20 μg per ml) at 289, 312·7 and 319 mμ obey Beer's law if **II** soln. (40 μg per ml) or **I** soln. (200 μg per ml), respectively, is used as reference. *Procedure*—Dissolve almost pure **II** (250 to 300 mg) in 0·05N-H_2SO_4 (50 ml), dilute a 5-ml aliquot to 50 ml with 0·05M-NaOH and further dilute 10-fold with 0·05M-NaOH. Measure the extinction at 289 and 319 mμ, or at 312·7 and 319 mμ, against **II** soln. (40 μg per ml). Calculate the amount of **I** and **II** by the usual method. Prepare almost pure **I** soln. containing 200 to 300 μg per ml in 0·05M-NaOH and measure the extinction at 319 mμ against **I** in 0·05M-NaOH (200 μg per ml). Dilute 10-fold and measure at 312·3 mμ. Calculate the amount of **I** and **II** by the empirical formula given.
K. SAITO

2939. **The longest wavelength band in the electronic spectra of polynuclear heterocyclic aromatics for analytical use.** C. Karr, jun. (Bureau of Mines, U.S. Dept. of the Interior, Morgantown, W. Va., U.S.A.). *Appl. Spectroscopy*, 1960, **14** (6), 146–153.—A list of 204 compounds is presented, arranged according to increasing values in mμ of the longest wavelength absorption band of the spectra. It is an extension of the previous list of 408 compounds, all of which contain 3 or more aromatic rings (*Ibid.*, 1959, **13**, 15; 40). The definition "aromatic" has here been extended to include heterocyclic compounds. E. G. CUMMINS

2940. **X-ray diffraction powder data for some 2,4,6-substituted 1,3,5-triazine compounds.** W. L. Baun (Wright Air Dev. Div., Wright-Patterson Air Force Base, Ohio, U.S.A.). *Appl. Spectroscopy*, 1960, **14** (6), 153–156.—Data are presented for 15 compounds. Methods of minimising preferred orientation in pure compounds, and for the analysis of mixtures and impure compounds, are discussed. The complexity of the substitution presents no problems. E. G. CUMMINS

2941. **Use of precipitation chromatography for the detection of some benzothiazole derivatives.** A. Popov and V. Gydeva (Inst. Chem., Bulg. Acad. Sci.). *Compt. Rend. Acad. Bulg. Sci.*, 1960, **13** (4), 411–414 (in Russian).—Methods of detecting accelerators in rubber (Popov *et al.*, *Ibid.*, 1959, **12**, 419) have been extended to the following compounds, *viz*, dibenzothiazol-2-yl disulphide (**I**),

benzothiazole-2-sulphon-diethylamide (**II**) and -cyclohexylamide (**III**), zinc salts of 2-mercaptobenzothiazole (**IV**), and 2-mercaptobenzimidazole (**V**). *Procedure*—Heat 0·1 to 0·2 g of the sample under reflux for 1 hr. with a mixture of 96% ethanol (45 ml) and conc. HCl (5 ml). Distil off most of the ethanol, leaving a residue of 10 to 20 ml. Dilute 5 to 10 ml of this soln. with H_2O (10 to 20 ml) and extract with benzene (10 ml). Pass the dried benzene layer through a column of $Bi(NO_3)_3$. An orange - yellow band indicates the presence of mercaptobenzothiazole, obtained by decomposition of **I**, **II** or **III**. Neutralise a further 1 to 2 ml with NaOH soln. (10%), heat to boiling and test the steam with litmus paper. **I** gives no reaction, and an alkaline reaction indicates **II** or **III**. Make a further portion alkaline with Na_2CO_3 and add sodium nitroprusside. A blue colour due to diethylamine indicates **II**, while **III** gives no reaction. As the zinc salts of **IV** are almost insol. in org. solvents they must be heated with acetic acid before extraction with benzene and passage through the column. A soln. of **V** in amyl acetate gives an orange - red band on a $Bi(NO_3)_3$ column, and a blue - green one on a $CoCl_2$ column.

J. W. PRICE

2942. **Determination of tetramethyl-lead and tetraethyl-lead in gasoline by mass spectrometry.** H. E. Howard, W. C. Ferguson and L. R. Snyder (Res. Dept., Union Oil Co. of Calif., Brea, U.S.A.). *Anal. Chem.*, 1960, **32** (13), 1814–1815.—The compounds named are determined by comparing the mass spectrum of the sample with the mass spectra of two synthetic blends having known concn. of tetramethyl- and tetraethyl-lead. Peak heights are measured at m/e^+ 253 and 295, respectively, and the results obtained on synthetic mixtures show excellent agreement. Appropriate corrections can be made in the presence of additives.

C. B. BAINES

2943. **Identification of carbazole in Wilmington petroleum through use of gas - liquid chromatography and spectroscopy.** R. V. Helm, D. R. Latham, C. R. Ferrin and J. S. Ball (Petrol. Res. Center, Bureau of Mines, U.S. Dept. of the Interior, Laramie, Wyo.). *Anal. Chem.*, 1960, **32** (13), 1765–1767.—Two methods are described for the separation and identification of carbazole, the first non-basic nitrogen compound to be isolated from a virgin petroleum. One of the methods was selected to minimise the possibility of thermal decomposition, and temperatures did not exceed 110°. The separation is carried out on a column of high-vacuum silicone grease supported on Chromosorb maintained at 250°. Mass, i. r. and u. v. spectroscopy were used to identify carbazole. C. B. BAINES

2944. **Simplified method for determining carbon, hydrogen and sulphur in solid and liquid fuels.** W. Radmacher and A. Hoverath (BrennstChem. Inst., Ruhrkohlen-Beratung G.m.b.H., Essen, Germany). *BrennstChemie*, 1960, **41** (10), 304–308.—The quartz combustion tube (*T*) comprises a volatilisation section (*V*) from which the combustible vapours are swept by a current of N through a sealed-in axial burner-nozzle (2 mm bore) into the combustion section, where they meet a current of O introduced through a side-tube. An external burner heats the nozzle to the ignition temp. so that a visible hot flame is formed, and complete combustion is assured by regulating the flow of O without any oxidant packing. When all the volatile products have been burned, the N current through *V* is replaced by O to complete the combustion of residual C, etc. The combustion products, after passing through a quartz-wool packing, enter the final part of *T*, which is packed with PbO and kept at 200°, by a sealed-on quartz jacket containing H_2SO_4 and $KHSO_4$. Volatile liquids are weighed and introduced into *V* in a small stoppered quartz tube. Special absorption vessels containing enough $Mg(ClO_4)_2$ and NaOH for several macro- or semi-micro determinations are connected to *T* through a silicone stopper (*cf. Ibid.*, 1960, **41**, 52). If S is to be determined, the procedure is as described above, but N is replaced by Ar as carrier gas, and the PbO packing is omitted, the final section of *T* being bent downward with a stoppered inlet at the elbow whereby condensed H_2SO_4 can be washed out for determination by the Seuthe method (DIN 51724). Agreement with the Sheffield method (C and H) and with DIN 51724 and 51768 (S in solid fuels and heavy oils) is good. A. R. PEARSON

2945. **Methods for the analysis and testing of coal and coke. Part 10. Arsenic in coal and coke.** British Standards Institution (2 Park Street, London, W.1). B.S. 1016: Part 10: 1960. 22 pp.—Two methods are described, a colorimetric method and a modified Gutzeit method.

2946. **Determination of the naphthalene content of town gas by the use of picric acid. Examination of some factors influencing the precipitation of naphthalene picrate.** G. Iványi and V. Róna (Res. Inst. for Heavy Chem. Ind., Veszprém, Hungary). *Magyar Kém. Lapja*, 1960, **15** (12), 568–571.—After a brief review of the literature, the results of model tests designed to assess the effect of various factors in the pptn. of naphthalene picrate from soln. of naphthalene and picric acid are described. The amount and morphological structure of the naphthalene picrate depend in reproducible fashion on the speed of pptn. The amount of naphthalene remaining in soln. after pptn. by saturated picric acid is independent of the excess of picric acid used, provided that the naphthalene concn. is initially <120 mg per 100 ml. Change of temp. (5° to 30°) has little effect on the reaction. An examination of the ppt. showed that, with a content of naphthalene <60 mg, the relative error caused by adsorption on the ppt. is $<0{\cdot}15\%$; best results are obtained when washing is omitted. Determinations of known amounts of naphthalene vapour by absorption in picric acid soln. were also carried out; under the conditions employed, results were in good agreement with the amounts taken. The findings of these investigations were used for modifying older methods for determining naphthalene contents of coke-oven and town gas. (17 references.)

B. VASZKO

2947. **Determination of anthracene. III (2). Errors in xylene distillation method.** Eiichi Funakubo, Hiroshi Taniguchi and Isao Kawanishi (Chem. Inst., Tech. Fac., Osaka Univ., Japan). *BrennstChemie*, 1960, **41** (10), 308–312.—It is estimated that, for samples of anthracene content $\ll 30\%$, the error rarely exceeds $\pm 0{\cdot}36\%$, and, for contents of 10%, $\pm 0{\cdot}45\%$. (*Cf.* Funakubo, *Ibid.*, 1960, **41**, 243.) A. R. PEARSON

2948. **Improved titrimetric method for determining sulphur trioxide in flue gases.** R. S. Fielder and C. H. Morgan (Central Electricity Res. Lab., Leatherhead, Gt. Britain). *Anal. Chim. Acta*,

portions of a mixture of ethanol and H_2SO_4 (100 ml) (filter the last portions through paper) and titrate with 0·1N-iodine till the soln. becomes yellow - green. (*ii*) *Paper chromatography*—Place the sample soln. (2 g in 10 ml of ethanol) on WF 1 paper impregnated with 10% 1-bromonaphthalene soln. in ethanol and develop by the descending technique with 80% acetic acid satd. with 1-bromonaphthalene. Locate the spots with a soln. of K_2PtI_6 (0·5 ml of 5% $PtCl_4$ in N-HCl mixed with 45 ml of 10% KI soln. and 5 ml of H_2O) (pale-yellow spots of dibenzyl sulphide and yellow or yellow - brown spots of disulphide and polysulphides on a pink background). (*iii*) *Polarography of dibenzyl tetrasulphide*—Dissolve the sample (0·5 g) in benzene - methanol (1:1) and dilute with H_2O to 50 ml; mix an aliquot (1 to 10 ml) with 2M-acetate soln. (13·6 g of cryst. Na acetate plus 5·8 ml of anhyd. acetic acid dissolved in 250 ml of methanol and diluted to 500 ml with benzene) (10 ml) and record the polarographic wave from −0·2 to −1·2 V *vs.* the mercury pool. J. ZÝKA

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C. B. BAINES

2943. **Identification of carbazole in Wilmington petroleum through use of gas-liquid chromatography and spectroscopy.** R. V. Helm, D. R. Latham, C. R. Ferrin and J. S. Ball (Petrol. Res. Center, Bureau of Mines, U.S. Dept. of the Interior, Laramie, Wyo.). *Anal. Chem.*, 1960, **32** (13), 1765–1767.—Two methods are described for the separation and identification of carbazole, the first non-basic nitrogen compound to be isolated from a virgin petroleum. One of the methods was selected to minimise the possibility of thermal decomposition, and temperatures did not exceed 110°. The separation is carried out on a column of high-vacuum silicone grease supported on Chromosorb maintained at 250°. Mass, i. r. and u. v. spectroscopy were used to identify carbazole. C. B. BAINES

2944. **Simplified method for determining carbon, hydrogen and sulphur in solid and liquid fuels.** W. Radmacher and A. Hoverath (BrennstChem. Inst., Ruhrkohlen-Beratung G.m.b.H., Essen, Germany). *BrennstChemie*, 1960, **41** (10), 304–308.—The quartz combustion tube (*T*) comprises a volatilisation section (*V*) from which the combustible vapours are swept by a current of N through a sealed-in axial burner-nozzle (2 mm bore) into the combustion section, where they meet a current of O introduced through a side-tube. An external burner heats the nozzle to the ignition temp. so that a visible hot flame is formed, and complete combustion is assured by regulating the flow of O without any oxidant packing. When all the volatile products have been burned, the N current through *V* is replaced by O to complete the combustion of residual C, etc. The combustion products, after passing through a quartz-wool packing, enter the final part of *T*, which is packed with PbO and kept at 200°, by a sealed-on quartz jacket containing H_2SO_4 and $KHSO_4$. Volatile liquids are weighed and introduced into *V* in a small stoppered quartz tube. Special absorption vessels containing enough $Mg(ClO_4)_2$ and NaOH for several macro- or semi-micro determinations are connected to *T* through a silicone stopper (*cf. Ibid.*, 1960, **41**, 52). If S is to be determined, the procedure is as described above, but N is replaced by Ar as carrier gas, and the PbO packing is omitted, the final section of *T* being bent downward with a stoppered inlet at the elbow whereby condensed H_2SO_4 can be washed out for determination by the Seuthe method (DIN 51724). Agreement with the Sheffield method (C and H) and with DIN 51724 and 51768 (S in solid fuels and heavy oils) is good. A. R. PEARSON

2945. **Methods for the analysis and testing of coal and coke. Part 10. Arsenic in coal and coke.** British Standards Institution (2 Park Street, London, W.1). B.S. 1016: Part 10: 1960. 22 pp.—Two methods are described, a colorimetric method and a modified Gutzeit method.

2946. **Determination of the naphthalene content of town gas by the use of picric acid. Examination of some factors influencing the precipitation of naphthalene picrate.** G. Iványi and V. Róna (Res. Inst. for Heavy Chem. Ind., Veszprém, Hungary). *Magyar Kém. Lapja*, 1960, **15** (12), 568–571.—After a brief review of the literature, the results of model tests designed to assess the effect of various factors in the pptn. of naphthalene picrate from soln. of naphthalene and picric acid are described. The amount and morphological structure of the naphthalene picrate depend in reproducible fashion on the speed of pptn. The amount of naphthalene remaining in soln. after pptn. by saturated picric acid is independent of the excess of picric acid used, provided that the naphthalene concn. is initially < 120 mg per 100 ml. Change of temp. (5° to 30°) has little effect on the reaction. An examination of the ppt. showed that, with a content of naphthalene < 60 mg, the relative error caused by adsorption on the ppt. is $< 0{\cdot}15\%$; best results are obtained when washing is omitted. Determinations of known amounts of naphthalene vapour by absorption in picric acid soln. were also carried out; under the conditions employed, results were in good agreement with the amounts taken. The findings of these investigations were used for modifying older methods for determining naphthalene contents of coke-oven and town gas. (17 references.)

B. VASZKO

2947. **Determination of anthracene. III (2). Errors in xylene distillation method.** Eiichi Funakubo, Hiroshi Taniguchi and Isao Kawanishi (Chem. Inst., Tech. Fac., Osaka Univ., Japan). *BrennstChemie*, 1960, **41** (10), 308–312.—It is estimated that, for samples of anthracene content $\leqslant 30\%$, the error rarely exceeds $\pm 0{\cdot}36\%$, and, for contents of 10%, $\pm 0{\cdot}45\%$. (*Cf.* Funakubo, *Ibid.*, 1960, **41**, 243.) A. R. PEARSON

2948. **Improved titrimetric method for determining sulphur trioxide in flue gases.** R. S. Fielder and C. H. Morgan (Central Electricity Res. Lab., Leatherhead, Gt. Britain). *Anal. Chim. Acta*,

1960, **23** (6), 538–540 (in English).—Sulphur trioxide [0 to 50 p.p.m. (v/v)] is determined in flue gases that also contain 2000 to 3000 p.p.m. (v/v) of SO_2. *Procedure*—Absorb the gases in a mixture of isopropyl alcohol and water (4:1) in the apparatus previously described (*cf.* Fielder *et al.*, *J. Inst. Fuel*, 1960, **33**, 84). To an aliquot (10 to 50 ml) of the soln., containing 50 to 500 μg of SO_3, add 5 drops of thoron soln. (0·04%) for each 20 ml and titrate the SO_4^{2-} with 0·0025M-$Ba(ClO_4)_2$, acidified to pH 2·5 to 4·0 with $HClO_4$, to a pink end-point.

H. N. S.

2949. **Determination of total phosphoric oxide and total boric oxide in detergent powders.** D. C. Cullum and D. B. Thomas (Colgate-Palmolive Ltd., 371 Ordsall Lane, Manchester, England). *Analyst*, 1960, **85**, 922–923.—The detergent powder (4 to 5 g) is first charred and then ignited to a white ash at 700° to 800° and the cooled residue is dissolved in 3N-HNO_3. The soln. is diluted with 3N-HNO_3 to a total vol. of ≮25 ml and boiled under reflux for ≮30 min. The cooled, electrically stirred liquid is adjusted by means of a pH meter to pH 3·5 to 4 with 3N-NaOH and then accurately to pH 4·5 with N-NaOH. A 10% $AgNO_3$ soln. (100 ml) is added and the liquid is titrated with N-NaOH to pH 6 (1 ml ≡ 0·0355 g of P_2O_5). After removal of the electrodes and stirrer the Ag_3PO_4 is collected by suction on two thicknesses of Whatman No. 42 filter-paper. To the filtrate are added 5 ml of 3N-HNO_3 and sufficient satd. NaCl soln. to precipitate all the excess of Ag^+. After filtration, the pH of the filtrate is adjusted to 5·5 with N- and then 0·1N-NaOH. After addition of a vol. of glycerol about half that of the soln., the liquid is titrated slowly to pH 8·5 with 0·1N-NaOH (1 ml ≡ 0·003484 g of B_2O_3). Quoted results indicate satisfactory accuracy.

A. O. Jones

2950. **Amperometric titration of some acid azo-dyes with benzidine solution as titrant.** M. Matrka and Z. Ságner (Res. Inst. Org. Synth., Pardubice-Rybitví, Czechoslovakia). *Chem. Prùmysl*, 1960, **10** (12), 638–639.—The precipitation reaction of benzidine with the sulphonic acid group of some acid azo-dyes was used for their determination; the polarographic current of the dye was used for the amperometric titration with the dropping-mercury electrode - S.C.E. system at −0·4 to −0·8 V. *Procedure*—Dissolve 0·00125 g of dye in 500 ml of H_2O. To 50 ml add 2·5N-HCl (5 ml), remove O with a stream of N and titrate with 0·025M-benzidine (dissolve 4·606 g of freshly distilled benzidine in 50 ml of 2·5N-HCl and dilute with H_2O to 1 litre; the soln. is stable in a brown bottle); after each addition of titrant the soln. must be bubbled with N for 1 min. One mole of sulphonic acid group reacts with 0·5 mole of benzidine. The errors in the titration of orange II (C.I. Acid Orange 7), Crystal Ponceau 6R (C.I. Acid Red 41) and brilliant yellow (C.I. Direct Yellow 4) did not exceed ±0·8% and the results were in good agreement with those by titanometric determination.

J. Zýka

2951. **Rapid identification of cellulose esters and ethers using paper chromatography.** E. B. Mano and L. C. O. Cunha Lima (Inst. Nacional de Tecnol., 82 Avenida Venezuela, Rio de Janeiro, Brazil). *Anal. Chem.*, 1960, **32** (13), 1772–1773.—A procedure is described for the identification of cellulose acetate, acetate-butyrate and nitrate, methyl-, ethyl-, hydroxyethyl- and carboxymethyl-cellulose, and cellulose. The sample is divided into 3 groups by its solubility in H_2O and acetone. The acetone extract contains the cellulose esters and ethyl cellulose, the aq. extract contains the remaining ethers and the residue is cellulose. *Test A*—A fragment of the sample is heated with H_3PO_4 and the vapour is passed through filter-paper moistened with aniline acetate soln. A yellow spot indicates cellulose nitrate, a pink spot indicates other derivatives. Acetate and acetate-butyrate are detected by paper chromatography of the acids after saponification. *Test B*—A fragment is warmed with benzene and H_2SO_4 until a yellow colour which turns reddish is produced. The soln. is cooled and layered with ethanol. A violet ring indicates ethyl cellulose, a blue or green ring indicates other derivatives. Tests *A* and *B* are made on each extract and on the residue, unless the sample is completely soluble in one of the solvents, when they are made on the original solid. Methyl- and hydroxyethyl-cellulose cannot be distinguished. Confirmatory tests are described.

G. Burger

2952. **Chromatographic estimation of sugars in wood-cellulose hydrolysates. A routine reflectance method.** J. E. Jeffery, E. V. Partlow and W. J. Polglase (Olympic Res. Div., Rayonier Inc., Shelton, Wash.). *Anal. Chem.*, 1960, **32** (13), 1774–1777.—A method is presented for the rapid routine determination of arabinose, xylose, galactose, glucose and mannose in wood-cellulose hydrolysates without interference from uronic acids. The hydrolysing acid is neutralised with $BaCO_3$, the slurry is filtered without washing and an aliquot of the filtrate is evaporated to 2 ml and made up to 5 ml with 0·25% aq. benzoic acid. This soln. is chromatographed on paper with butyl acetate - pyridine - ethanol - water (8:2:2:1). The colour is developed by dipping the chromatogram in a soln. of 0·7% aniline and 1·4% chloroacetic acid in ethyl ether. Before and after colour development the paper is hung in a humidity cabinet and then dried. Reflectance measurements at 375 mμ are converted into K/S values [$= (R-1)^2/2R$, where R is the % reflectance] and the concn. of the sugars are found from standard curves of K/S values *vs.* concn.

G. Burger

2953. **Determination of pentosans in chemical wood pulps.** I. Grobelny and W. Illukowicz (Polish Pulp and Paper Res. Inst., Łódź). *TAPPI*, 1960, **43** (7), 244A–246A.—Three methods are compared—(*a*) distillation with 13% HCl or (*b*) with 20% HBr, followed by determination of furfuraldehyde by bromination, and (*c*) distillation with 3·85N-HCl, followed by determination of furfuraldehyde with orcinol. Comparison of the results shows that conditions of hydrolysis and distillation have an appreciable effect. Method (*c*) gave the most accurate and best reproducible results, and is recommended for research work, but its cost is high, a photometer is necessary, and the low stability of orcinol in HCl is a disadvantage. The recoveries of furfuraldehyde and xylose are 98·5% and 94·5%, respectively. Method (*a*) gives good reproducibility after double distillation with 13% HCl. The method and apparatus are simple and inexpensive, but the recovery of furfuraldehyde and xylose is lower than in method (*c*). The method is recommended for mill control. Method (*b*) gave satisfactory results with high pentosan contents, but it has the disadvantage of foaming and carry-over during distillation and is difficult to use with small amounts of pentosans.

R. Aicher

2954. **Tentative Textile Standard No. 64. Determination of oils, fats and waxes in wool.** (Textile Inst., 10 Blackfriars Street, Manchester, England). *J. Text. Inst. Proc.*, 1960, **51** (11), P657–P660.—A conditioned and weighed specimen of wool is extracted in a Soxhlet extractor with ethyl ether under defined conditions. The filtered ether solution is evaporated and the residue dried and weighed. Its weight is expressed as a percentage of the original conditioned weight of the specimen, or of the dry-weight of the specimen, or of the dry-weight of the specimen corrected by adding the appropriate official regain (correct invoice-weight).

O. M. WHITTON

2955. **Tentative Textile Standard No. 65. Determination of oils, fats and waxes in cotton.** (Textile Inst., 10 Blackfriars Street, Manchester, England). *J. Text. Inst. Proc.*, 1960, **51** (11), P660–P662.—A conditioned and weighed specimen of known moisture regain is extracted in a Soxhlet extractor under defined conditions. The solvent is evaporated and the specimen dried and weighed. The result is expressed as a percentage of the original air-dry weight, or of the dry-weight. The method applies to the removal and estimation of oil, fat, wax or fatty acid in raw, scoured or bleached cotton, and in cotton containing a size or finish of tallow, spermaceti and other natural fats or waxes, mineral oil, paraffin wax and fatty acids derived from soaps by hydrolysis. It does not apply to materials containing oxidised unsaturated oils, waterproofing or softening preparations derived from cetylpyridinium halides, rot-proofing agents or synthetic resins.

O. M. WHITTON

2956. **Rapid method for determination of total sulphur in viscose.** J. F. Crow, J. P. Dux and L. H. Phifer (American Viscose Corp., Marcus Hook, Pa.). *TAPPI*, 1960, **43** (7), 620–622.—The viscose (0·1 to 0·2 g) in a combustion boat is heated in a Dietert-Detroit Varitemp combustion furnace in a stream of oxygen. The boat is advanced in three distinct stages and remains in the hottest zone for <15 min. The effluent gases are bubbled through 30% H_2O_2 soln., and at the end of the reaction the bubbler soln. is titrated with standard 0·005M-$Ba(ClO_4)_2$, with 2 drops of thoron as indicator. The colour change is sharp from pale yellow to pale pink. A duplicate analysis takes 30 min. and the precision of the method is good.

R. AICHER

2957. **Paper chromatography in the fat field. XLIII. Analysis of alkyd resins.** H. P. Kaufmann and F. J. Büscher (Dtsch. Inst. f. Fettforsch., Münster, Westf., Germany). *Fette, Seif., Anstrichmitt.*, 1960, **62** (12), 1141–1143.—Alkyd resins (150 to 200 μg) were applied at the starting point and saponified with 2% KOH in glycerol; the paper strips were pressed between glass plates and maintained at 90° for 1·5 to 2 hr. After cooling the strips were set aside in 2N-HCl for 30 min., washed until neutral, and dried. The chromatogram was developed with glacial acetic acid - methyl cyanide - undecane, and the separated fatty acids were detected with Cu acetate and ethanolic dithiooxamide soln. The photometric method of Seher (*Anal. Abstr.*, 1960, **7**, 2999) was used for the determination. A blank was carried out on the oils used for the manufacture of the resins.

I. DICKINSON

2958. **Determination of additives in polyethylene by absorption spectroscopy.** H. L. Spell and R. D. Eddy (Dow Chemical Co., Freeport, Tex.). *Anal. Chem.*, 1960, **32** (13), 1811–1814.—A procedure is described for the solvent extraction of additives from polyethylene pellets by shaking at room temp. followed by their determination in concn. up to 500 p.p.m. with a precision of ± 2 p.p.m. Ionol (2,6-di-t-butyl-*p*-cresol) and Santonox [4,4′-thiobis-(6-t-butyl-*m*-cresol)] are extracted with CS_2 in 1·75 hr. and 2·5 hr., respectively, or in 50 hr. and 76 hr. with iso-octane when interfering substances are present. The CS_2 soln. are scanned from 7·8 to 9·3 μ, and the iso-octane soln. from 250 to 350 mμ. Oleamide is extracted with CCl_4 in 21 hr. and the soln. is scanned from 5·4 to 6·2 μ or from 2·6 to 3·4 μ if interfering materials are present.

G. BURGER

2959. **Determination of terminal epoxides, particularly styrene oxide.** B. D. Sully (A. Boake Roberts & Co. Ltd., London, England). *Analyst*, 1960, **85**, 895–897.—To 40 ml of aq. $MgSO_4$ - $Na_2S_2O_3$ soln. (12·5 g of cryst. $MgSO_4$ and 12·5 g of cryst. $Na_2S_2O_3$ in 100 ml) are added 20 ml of ethanol and 1 ml of screened phenolphthalein - methyl red indicator soln. (1 g of phenolphthalein, 0·02 g of methyl red and 0·01 g of methylene blue in 100 ml of ethanol). The pH is adjusted to 8·25 (the higher end-point of the indicator) with 0·1N-NaOH and the weighed sample containing 0·5 to 0·7 g of styrene oxide is added. The mixture is boiled under reflux for 15 min., cooled, diluted with 30 ml of water and titrated with 0·2N-acetic acid. To ensure complete dissolution of the pptd. $Mg(OH)_2$, an excess of $\simeq 5$ ml of 0·2N-acetic acid is added and the excess is titrated with 0·1N-NaOH.

A. O. JONES

See also Abstracts—**2782**, Hg in cellulose. **2973**, Paper chromatography of phenols. **3007**, Identification of org. compounds. **3052**, Disinfectant activity of quaternary ammonium compounds. **3066**, Anti-foaming device for Kjeldahl digestion.

4.—BIOCHEMISTRY

INCLUDING DRUGS, FOOD, SANITATION, AGRICULTURE

Biological fluids, animal and vegetable tissues

2960. **Use of sulphosalicylic acid in the polarographic determination of chlorides in biological material.** M. Bartík and J. Kupka (Inst. of Chem. and Phys., Veterinary Fac., Košice, Czechoslovakia). *Coll. Czech. Chem. Commun.*, 1960, **25** (12), 3391–3393 (in German).—Protein is pptd. by sulphosalicylic acid, which then forms a suitable supporting electrolyte for the determination of Cl^- and also shifts the oxygen wave to a more negative potential ($E_{\frac{1}{2}} = -0{\cdot}19$ V), so that it does not interfere with the Cl^- wave ($E_{\frac{1}{2}} = +0{\cdot}22$ V). Removal of oxygen is then unnecessary. *Procedure*—Add 1 ml of the biological fluid or extract (containing up to 300 mg of NaCl per 100 ml) to 20% sulphosalicylic acid soln. (2 ml). Dilute to 10 ml with water, centrifuge, and record the polarogram.

A. J. BENNETT

2961. **Polarographic determination of nitrate in biological material.** M. Bartík and J. Kupka (Inst. of Chem. and Phys., Veterinary Fac., Košice, Czechoslovakia). *Coll. Czech. Chem. Commun.*, 1960, **25** (12), 3356–3362 (in German).—If much

protein is present in the material it is pptd. with sulphosalicylic acid. On the addition of conc. H_2SO_4 the nitrate reacts with the sulphosalicylic acid to give a reducible nitro-compound, which yields a polarographic wave proportional to the original nitrate concn. *Procedure*—To 0·5 ml of blood or serum add 0·5 ml of water. Mix with 20% sulphosalicylic acid (1 ml) and centrifuge. Add 1 ml of the supernatant liquid to 2 ml of conc. H_2SO_4 and shake. After cooling the soln. add 2 ml of water and record the polarogram ($E_{\frac{1}{2}} = -0{\cdot}08$ V). This procedure is not applicable to some materials, *e.g.*, urine and the contents of digestive organs, because the nitrate then also reacts with other aromatic compounds present. With such materials, of low protein content, the sulphosalicylic acid is therefore replaced by phenol and the nitration is carried out as described above, the nitrophenol formed having a $E_{\frac{1}{2}}$ value of $+0{\cdot}03$ V.

A. J. Bennett

2962. **Rapid method for estimation of carbon monoxide in blood.** M. Henderson and G. H. Apthorp (St. Bartholomew's Hosp., London), *Brit. Med. J.*, 1960, 1853–1854.—A rapid modification of the re-breathing method (Sjöstrand, *Acta Physiol. Scand.*, 1948, **16**, 201) is described.

H. F. W. Kirkpatrick

2963. **Colorimetric micro-determination of calcium in serum.** P. Trinder (Biochem. Dept., Central Lab., Royal Infirmary, Sunderland, Co. Durham, England). *Analyst*, 1960, **85**, 889–894.—The serum (0·2 ml) is treated with a naphthohydroxamic acid reagent (prep. described) and after 30 min. is centrifuged at 3500 r.p.m. for 5 min. The supernatant liquid is removed and the ppt. is suspended in 1 ml of alkaline EDTA soln. (2 g of the disodium salt in 1 litre of 0·1N-NaOH). The ppt. is dissolved in boiling water, and 3 ml of the colour reagent [60 g of $Fe(NO_3)_3.9H_2O$ and 15 ml of conc. HNO_3 in 1 litre] is added to the cold soln. A blank determination is made with omission of the serum. The extinction of the sample soln. is measured against the blank soln. at 450 mμ or with a Chance OB10 filter, and the Ca content of the serum is calculated by comparing the extinction with that of standard Ca soln. Recovery ranged from 100 to 102% in the absence of other substances and from 100 to 103% in their presence.

A. O. Jones

2964. **Micro-determination of serum copper.** R. M. Summers (Psychiatric and Psychosomatic Res. Lab., V. A. Hospital, Houston, Tex., U.S.A.). *Anal. Chem.*, 1960, **32** (13), 1903–1904.—The adaptation of the oxalyldihydrazide - acetaldehyde method of Stark and Dawson (*Anal. Chem.*, 1958, **30**, 191) to an automatic procedure is described. Manual procedures are less precise, more tedious and more time consuming.

D. C. Armsworth

2965. **Paper-chromatographic separation of a micro amount of inorganic phosphate in urine.** Tadakatsu Iwanaga (Fac. of Engng, Gifu Univ., Naka-machi). *J. Chem. Soc. Japan, Pure Chem. Sect.*, 1960, **81** (6), 984–986.—When urine (pH 5·4) (0·0025 ml) is developed with a mixture of methanol, 85% formic acid and water (16:3:1), PO_4^{3-} (R_F 0·70) are separated from Cl^- (R_F 0·6). SO_4^{2-} (0·4), AsO_4^{3-} (0·2) and As^{3+} (0·08). The PO_4^{2-} are extracted with 8N-aq. NH_3 or the spot is ashed with the paper and the P determined photometrically by the molybdenum blue method.

K. Saito

2966. **Determination of small amounts of mineral in bone, cartilage and muscle by X-ray absorption.** G. P. Vose (The Nelda Childers Stark Lab. for Human Nutritional Res., Texas Woman's Univ., Denton, U.S.A.). *Microchem. J.*, 1960, **4** (4), 537–549.—The material is partially dried, pulverised in a Waring Blendor, and dried to constant weight. Portions are added to 10% HCl containing 0·08% of a non-ionic surface-active agent, and the soln. are filtered after 1 hr. The absorption of *K*-capture X-radiation from a ^{55}Fe source by 0·04 ml of the soln. is measured by a Geiger tube and a Tracerlab SC19 binary scaler. The absorption of the soln. is a function of its mineral concn. Specimens of 50 mg can be analysed with a probable error of $<2\%$.

A. J. Bennett

2967. **Determination of methanol in biological fluids.** C. D. Hough (Public Health Lab., P.O. Box 138, Lusaka, N. Rhodesia). *Analyst*, 1960, **85**, 921–922.—Three Cavett flasks are each charged with 3 ml of H_2SO_4 (1:2) and 1 ml of $KMnO_4$ soln. (0·3% w/v) and the cups with 0·1 ml of satd. aq. K_2CO_3 soln. To one cup is added 0·5 ml of the blood or urine sample and to the others 0·1 and 0·5 ml of standard methanol soln. (12·5 ml of methanol per litre, diluted 20-fold as required). The stoppered flasks are incubated at 37° for 4 hr., then to the cooled contents are added satd. aq. Na_2SO_3 soln. until the $KMnO_4$ is decolorised. Chromotropic acid (0·1 ml of a 0·5% w/v soln. of the sodium salt, freshly prepared) and conc. H_2SO_4 (4 ml) are added and the flasks are heated in boiling water for 10 min. The cooled liquids are diluted to 10 ml and the colour is measured against a reagent blank with an Ilford No. 626 filter. The standards correspond to concn. of 10 and 50 mg of methanol per 100 ml of sample. Beer's law is obeyed up to a concn. of 70 mg per 100 ml; above this concn. a smaller sample should be used. If formaldehyde is suspected, a replicate determination is made without the $KMnO_4$ and the result is deducted. Ethanol does not interfere.

A. O. Jones

2968. **Oscillopolarographic study of barbiturates.** J. Prokeš and F. Vorel (Lab. Toxicol. and Forensic Med., Med. Fac., Charles' Univ., Prague, Czechoslovakia). *Chem. Zvesti*, 1960, **14** (11–12), 818–822.—The incisions on the curves $dE/dt = f(E)$ were used for a semi-quantitative determination of barbiturates in urine. Proteins, which interfere, are removed by paper chromatography. *Procedure*—Evaporate the acid ethereal extract of 10 ml of urine, dissolve the residue in ethanol and chromatograph on Whatman No. 3 paper with acetone. Extract the paper for 5 min. with 0·2M-Na_2HPO_4 (2 ml) on a water bath, transfer the soln. to a polarographic cell and measure the depth of the incision on the polarogram. Compare with a calibration curve. The method is suitable for the determination of 1×10^{-4}M to 4×10^{-4}M soln. of barbituric acid, barbitone, phenobarbitone, amylobarbitone, pentobarbitone and hexobarbitone. Thiamine interferes and cannot be separated chromatographically.

J. Žýka

2969. **Accurate method for the determination of chloramphenicol and its metabolites in blood, tissue homogenates and urine, and a study of the effect of blood-cell number on chloramphenicol concentration in serum.** Motohiro Maruyama and Yoshio Suzuki (Takamine Lab., Sankyo Co., Ltd., Shinagawa, Tokyo). *Ann. Rep. Takamine Lab.*, 1958, **10**, 158–177.—Whole blood is diluted with phosphate

buffer (0·2M, pH 6·0), extracted with ethyl acetate, washed with $NaHCO_3$ soln. and evaporated to dryness. The residue is extracted with water, rendered alkaline and treated with $Na_2S_2O_4$. The product is diazotised and coupled with 'Tsuda' reagent (β-diethylaminoethyl-α-naphthylamine oxalate). Chloramphenicol is assayed by measurement of the extinction at 560 mμ. The results are not affected by purines, pyrimidines, folic and *p*-aminobenzoic acids and D-(−)-threo-2-amino-1-(*p*-nitrophenyl)propane-1,3-diol. In 1 ml of blood, 0·5 μg of chloramphenicol can be determined. Blood serum is diluted with water and deproteinised with H_3PO_4. The total nitro-compounds are assayed in the supernatant liquid by a similar procedure to that for chloramphenicol. S. NATORI

2970. **Chromatography of hydroxyflavans on carboxymethylcellulose.** R. Neu (Firma Dr. Willmar Schwabe G.m.b.H., Karlsruhe-Durlach, Germany). *J. Chromatography*, 1960, **4** (6), 489–493 (in German).—Carboxymethylcellulose (**I**) is superior to polyamide for the purification and separation of flavones, flavanones, flavanols and their glycosides. Polymeric components of plant extracts are irreversibly adsorbed, but (+)-catechin and (−)-epicatechin are not. Pure compounds can be isolated from plant extracts by adsorption on packed columns of **I** followed by elution with acetone, water or methanol-water, and separations of flavanols from their dihydro-derivatives, and chalcones from flavanones, can be achieved by successive elution with solvents containing decreasing amounts of water.
W. T. CARTER

2971. **Quantitative determination of oats lichenin.** E. Letzig (Dtsch. Akad. d. Landwirtschaftswissenschaften, Berlin). *Nahrung*, 1960, **4** (9), 832–845.—For quant. analysis, the oats are finely crushed and kept at 150° for 1 hr. to inactivate the lichenin-decomposing enzymes, then boiled with water and digested with pepsin - HCl and pancreatin, and centrifuged. The lichenin is pptd. with ethanol - water (1:1), then hydrolysed with acid, and the resulting glucose is determined. Results of various samples are given. The lichenin content varies from 2·59 to 3·37%. I. DICKINSON

2972. **Analysis of extracts of *Atractylis gummifera* L. I. Chromatographic separation and identification of [potassium] atractylate and atractyligen on silica.** G. Fassina (Ist. di Farmacol., Univ. Padova, Italy). *Boll. Soc. Ital. Biol. Sper.*, 1960, **36** (24), 1417–1418.—The separation of the glycoside, K atractylate (**I**), from the aglycone, atractyligen (**II**), in extracts of *Atractylis gummifera* L., is effected chromatographically on thin layers of silica gel deposited on glass plates (18 cm × 12 cm) by Stahl's method (*ChemikerZtg*, 1958, **82**, 323). The chromatograms are developed by the ascending technique with n-butanol - methanol - H_2O (8:1:1) or n-propanol - xylene - H_2O (7:2:1) for 30 to 90 min. till the solvent front has risen 10 to 12 cm. Solvent is removed in warm air and the plates are sprayed with conc. H_2SO_4. **I** and **II** in the cold develop a salmon colour and show an intense golden-yellow fluorescence in Wood's light. After heating at 100° the colour changes to violet or blue - grey according to the amount of heating (5 to 10 min.). Other substances present either do not react or develop yellow colours in the cold and show a greenish-yellow fluorescence. The R_F values for **I** and **II** with the butanol mixture are 0·17 and 0·57 and, with the propanol mixture, 0·16 and 0·61, respectively.

II. Colorimetric determination of potassium atractylate and atractyligen. G. Fassina. *Ibid.*, 1960, **36** (24), 1419–1421.—**I** and **II** give dichroic colours, red by transmission, with yellow fluorescence in Wood's light, with *p*-methylaminobenzaldehyde (**III**) in H_2SO_4. The colour is not extracted by $CHCl_3$, light petroleum, benzene or xylene, but changes to blue on dilution with alcohol (methanol, ethanol, propanol or butanol), the intensity increasing with the chain length of the alcohol. Ether or acetone changes the colour to maroon. For the determination, add to the test soln. (0·1 ml) 1·9 ml of reagent [0·125 g of **III** dissolved in the cold in 100 ml of H_2SO_4 - H_2O (13:7)]. Heat on a boiling-water bath for 15 min. and add 3 ml of 96% ethanol. Measure the extinction at 570 mμ after 1 hr. The method is applicable over the range 2 to 60 μg of **I** and 1 to 30 μg of **II**, respectively, per ml of final reaction-mixture.
J. I. M. JONES

2973. **Supplementary data for the paper-chromatographic separation and identification of phenol derivatives and related compounds of biochemical interest using a "reference system."** L. Reio (Wenner-Gren Inst., Dept. of Metabolic Res., Univ. Stockholm, Sweden). *J. Chromatography*, 1960, **4** (6), 458–476 (in English).—The system previously described (*cf. Anal. Abstr.*, 1959, **6**, 1863) is extended to include a further 270 compounds. R_F values and colour reactions are given for monohydric, dihydric and trihydric phenols, naphthalene and quinoline derivatives, substituted benzoic and phthalic acids, aromatic, heterocyclic and aliphatic amino-acids, indole derivatives and biologically active nitrogen compounds. W. T. CARTER

2974. **Isolation and analysis of phosphatides and glycolipids.** H. Wagner (Inst. Pharm. Arzneimittellehre, Univ. München, Germany). *Fette, Seif., Anstrichmitt.*, 1960, **62** (12), 1115–1123.—Progress made in the detection, isolation and identification of new compounds by thin-layer and paper chromatography and application of the Craig distribution (Hörhammer *et al.*, *Biochem. Z.*, 1960, **332**, 269) and i.r. spectroscopy is reported. (64 references.)
I. DICKINSON

2975. **Modified method of quantitative determination of hippuric acid in urine.** G. Vassileff. *Compt. Rend. Acad. Bulg. Sci.*, 1960, **13** (6), 685–688 (in English).—Hippuric acid in urine (20 ml) is salted out with 5 to 6 g of NaCl from a strongly acid soln. (3 to 4 ml of 25% H_2SO_4) and extracted into ethyl ether. The ether is freed from H_2SO_4 by washing with satd. NaCl soln. and evaporated. The residue is dissolved in hot water and titrated with 0·1N-NaOH, with phenolphthalein as indicator. The determination takes 2 to 3 hr. and the errors are ≯2%. The method is superior to those commonly used in Quick's test. J. P. STERN

2976. **Ion-exchange chromatography of amino-acids. Study of effects of high pressures and fast flow rates.** P. B. Hamilton (A. I. du Pont Inst. of the Nemours Foundation, Wilmington 99, Del.). *Anal. Chem.*, 1960, **32** (13), 1779–1781.—Ion-exchange chromatography of amino-acids has been studied in glass columns at flow rates up to 0·17 cm per sec. and pressures up to 600 p.s.i. The columns were packed with Dowex 50-X8 or 50W-X8 resin beads closely graded for size. The effect of flow rate and particle size on resolution is discussed. The construction of the columns and couplings is described. G. BURGER

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2979. **Colorimetric and electrophoretic techniques to determine glutamic acid in the presence of γ-aminobutyric acid, with a reference to the estimation of the storage condition of wheat.** P. Linko (Dept. of Flour and Feed Milling Industries, Kansas Agric. Exp. Sta., Manhattan, U.S.A.). *Suomen Kem.*, 1960, **33** (7–8), 145–149 (in English).—To determine the glutamic acid decarboxylase activity of wheat, grind the sample (1 g) for 2 min. and immediately shake for 30 min. with 0·01M-glutamic acid (**I**) in M/15-phosphate buffer (pH 5·8) (5 ml) at 27°. Centrifuge at 1400 *g* for 15 min., dilute 2 ml of the supernatant liquid to 10 ml with 95% ethanol, and centrifuge again for 5 min. Dilute suitably and determine the concn. of **I** colorimetrically, or examine for **I** and γ-aminobutyric acid (**II**) electrophoretically. *Colorimetric procedure*—Shake the soln. (0·5 ml, containing <5 μmoles of amino-acid in H_2O or 70% ethanol) with a 2·5% soln. of citric acid in 95% ethanol (1 ml), an NH_3-free 80% (w/w) soln. of phenol in 95% ethanol (1 ml), NH_3-free pyridine (1 ml) containing 2% of a 0·01M aq. soln. of NaCN, and a 5% soln. of ninhydrin in 95% ethanol (0·2 ml). Incubate at 60° for 30 min., dilute to 100 ml with 60% ethanol and immediately measure the extinction at 570 mμ. **II** gives little colour in this reaction. *Electrophoretic procedure*—Apply the soln. (50 μl, containing <0·1 μmole of amino-acid) as a narrow streak to a strip of filter-paper and submit to electrophoresis in 0·025M-phthalate buffer (pH 4·0) for 90 min. at a potential gradient of 14 V per cm. Dry the strip at 60° and treat with ninhydrin and copper reagents. **I** and **II** are well separated from each other, and from lysine, arginine, glycine, alanine and aspartic acid. A. R. ROGERS

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fibrinogen is read from a calibration chart prepared from the results of micro-Kjeldahl determinations. Haemolysed plasma is unsuitable for this method. If required, the reading can be taken after 10 min. when about 95% of the maximum turbidity is formed. H. F. W. KIRKPATRICK

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2986. **Studies of cholesterol biosynthesis. I. The identification of desmosterol in serum and tissues of animals and man treated with MER-29 [triparanol]. [Determination of desmosterol.]** A. Avigan, D. Steinberg, H. E. Vroman, M. J. Thompson and E. Mosettig (Lab. of Cell. Physiol., Nat. Heart Inst., Bethesda, Md., U.S.A.). *J. Biol. Chem.*, 1960, **235** (11), 3123–3126.—Desmosterol gives a lower colour yield than cholesterol in the Liebermann - Burchard reaction when measured at 635 mμ. A colorimetric method, based on this, is described for the determination of desmosterol and cholesterol in mixtures of the two. The mixed sterols are pptd. as digitonides which, after being washed, are decomposed with pyridine at 100° for 10 min. The mixture of sterols is then extracted with ethyl ether and the colour reaction is carried out. After 35 min. the extinction is measured at 635 mμ; this gives the amount of cholesterol. The extinction is then determined at 420 mμ after 90 min. to give the amount of desmosterol. J. N. ASHLEY

2987. **Detection of non-ketonic steroids on paper chromatograms.** D. Kupfer, E. Forchielli, M. Stylianou and R. I. Dorfman (Worcester Foundn. for Exp. Biol., Shrewsbury, Mass., U.S.A.). *J. Chromatography*, 1960, **4** (6), 500–501 (in English).—The method depends on the oxidation of hydroxyl groups to ketonic functions that can form coloured products with Zimmermann reagent. The dried chromatograms are dipped in an 0·25% soln. of CrO_3 in 90% acetic acid and heated at 90° for 15 min. They are then dipped in 2·5N-ethanolic KOH, blotted thoroughly, and dipped in 2% ethanolic *m*-dinitrobenzene soln. and heated at 60° for 1 to 2 min. The limits of detection are 20 to 40 μg per sq. cm of paper. W. T. CARTER

2988. **Determination of individual 17-oxosteroids by gradient elution chromatography.** D. F. Johnson, E. Heftmann and D. Francois (Nat. Inst. of Arthritis and Metabolic Diseases, U.S. Dept. of Health, Education and Welfare, Bethesda, Md., U.S.A.). *J. Chromatography*, 1960, **4** (6), 446–450 (in English).—Eight urinary 17-oxosteroids are separated on a column of silica gel containing 25% (w/w) of water and 1·75% of methanol by the gradient elution technique with a mixture of water-saturated light petroleum and dichloromethane. The steroids in the separated fractions are identified by paper chromatography, and determined colorimetrically by the M.R.C. method (*Lancet*, 1951, **261**, 585). The effects of varying the composition of the stationary phase are reported, and the role of water in the separation is discussed. W. T. CARTER

2989. **The acid-catalysed solvolysis of dehydroepiandrosterone sulphate and its significance in the examination of urinary 17-oxosteroids. [Determination of dehydroepiandrosterone sulphate.]** L. Segal, B. Segal and W. R. Nes (Dept. of Chem., Clark Univ., Worcester, Mass., U.S.A.). *J. Biol. Chem.*, 1960, **235** (11), 3108–3111.—A 24-hr. sample of urine is neutralised with 5% aq. Na_2CO_3. One-third of the sample is made 3M with respect to NaCl and is extracted once with ethyl acetate (2 vol.); the organic phase is dried with anhyd. Na_2SO_4 (6 g per 100 ml of soln.). The dried and filtered extract is treated with sufficient H_2SO_4 in ethyl acetate to make the final concn. 2×10^{-4}M in H_2SO_4. The soln. is kept at 30° for 16 to 20 hr., then extracted once with N-NaOH (100 ml) and twice with water (100 ml). The organic layer is dried with anhyd. Na_2SO_4 and evaporated *in vacuo* at <40°. The residue is transferred with benzene (total vol. 4 ml) to a column (9 mm diam.) of Al_2O_3 (6 g) de-activated with water (4%). The column is eluted with a mixture of ethanol and benzene, starting with 0·2% and ending with 2% of ethanol in benzene. The dehydroepiandrosterone (androst-5-en-17-one) was determined in the eluates by the Zimmermann reaction. J. N. ASHLEY

2990. **Spectrophotometric determination of selenium in steroids.** L. J. Throop (Mead Johnson and Co., Evansville, Ind., U.S.A.). *Anal. Chem.*, 1960, **32** (13), 1807–1809.—The Se is separated from the organic compound with Raney nickel, oxidised to H_2SeO_3 with HNO_3, then converted with *o*-phenylenediamine into 1,2,3-benzoselenodiazole, the extinction of which is measured at 330 mμ. *Procedure*—Place the sample (containing 20 to 100 μg of Se) in a flask, dissolve it in 50 ml of ethanol and add about 300 mg of fresh Raney nickel prepared by the method of Drake (*Org. Synth.*, 1941, **21**, 15). Heat the soln. under reflux for 30 min., cool and decant the ethanol from the nickel, which is then washed by decantation with 30 ml of ethanol. Fit an air condenser and add 4 ml of HNO_3 dropwise with care. Warm the soln. to dissolve the nickel, cool and add 10 ml of H_2O. Boil the soln., cool and add 20 ml of H_2O and 10 mg of *o*-phenylenediamine dihydrochloride. Set aside for 15 min., then adjust the pH to $2·5 \pm 0·1$ with aq. NH_3. Transfer to a separating-funnel and extract with $CHCl_3$ (2×10 ml). Dry the $CHCl_3$ extract with anhyd. Na_2SO_4 and dilute to 25 ml with $CHCl_3$. Measure the extinction at 330 mμ against a $CHCl_3$ blank. Run a blank determination on the Raney nickel and subtract the value from the sample reading: Se (μg) = (extinction × 0·432 × 1·1)/(791 × 4). D. C. ARMSWORTH

2991. **Micro-method for amylase determinations using 0·05 ml of blood.** I. T. Beck, E. Pinter, R. D. McKenna and H. Griff (McGill Univ., Montreal, Canada). *Canad. Med. Ass. J.*, 1960, **82** (3), 152–153.—A modified Somogyi method is described. The sample of blood (0·05 ml) is added to 0·95 ml of 4% Na citrate soln. and is centrifuged, and 0·2 ml of the supernatant liquid is withdrawn for the amylase determination. The remaining 0·8 ml is treated with 1 ml of pptg. reagent (40 ml of water, 5 ml of 10% sodium molybdate soln. and 5 ml of 0·67N-H_2SO_4) and centrifuged for 5 min.; 1 ml of the supernatant liquid is used for the determination

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2985. **Oscillographic polarography of nucleic acids.** E. Paleček (Biophys. Inst., Acad. Sci., Brno, Czechoslovakia). *Chem. Zvesti*, 1960, **14** (11–12), 798–805.—Adenine, guanine, cytosine, 5-methylcytosine, thymine, uracil, hypoxanthine, xanthine and uric acid were studied oscillopolarographically in H_2SO_4, KCl, NaOH and ammonium formate. Examples are given for the detection and determination of these compounds in 10^{-5}M to 10^{-6}M soln., alone or in mixtures. The highly polymerised deoxyribonucleic acids and some of their degradation products were also studied. J. ZÝKA

2986. **Studies of cholesterol biosynthesis. I. The identification of desmosterol in serum and tissues of animals and man treated with MER-29 [triparanol]. [Determination of desmosterol.]** A. Avigan, D. Steinberg, H. E. Vroman, M. J. Thompson and E. Mosettig (Lab. of Cell. Physiol., Nat. Heart Inst., Bethesda, Md., U.S.A.). *J. Biol. Chem.*, 1960, **235** (11), 3123–3126.—Desmosterol gives a lower colour yield than cholesterol in the Liebermann - Burchard reaction when measured at 635 mμ. A colorimetric method, based on this, is described for the determination of desmosterol and cholesterol in mixtures of the two. The mixed sterols are pptd. as digitonides which, after being washed, are decomposed with pyridine at 100° for 10 min. The mixture of sterols is then extracted with ethyl ether and the colour reaction is carried out. After 35 min. the extinction is measured at 635 mμ; this gives the amount of cholesterol. The extinction is then determined at 420 mμ after 90 min. to give the amount of desmosterol. J. N. ASHLEY

2987. **Detection of non-ketonic steroids on paper chromatograms.** D. Kupfer, E. Forchielli, M. Stylianou and R. I. Dorfman (Worcester Foundn. for Exp. Biol., Shrewsbury, Mass., U.S.A.). *J. Chromatography*, 1960, **4** (6), 500–501 (in English).—The method depends on the oxidation of hydroxyl groups to ketonic functions that can form coloured products with Zimmermann reagent. The dried chromatograms are dipped in an 0·25% soln. of CrO_3 in 90% acetic acid and heated at 90° for 15 min. They are then dipped in 2·5N-ethanolic KOH, blotted thoroughly, and dipped in 2% ethanolic *m*-dinitrobenzene soln. and heated at 60° for 1 to 2 min. The limits of detection are 20 to 40 μg per sq. cm of paper. W. T. CARTER

2988. **Determination of individual 17-oxosteroids by gradient elution chromatography.** D. F. Johnson, E. Heftmann and D. Francois (Nat. Inst. of Arthritis and Metabolic Diseases, U.S. Dept. of Health, Education and Welfare, Bethesda, Md., U.S.A.). *J. Chromatography*, 1960, **4** (6), 446–450 (in English).—Eight urinary 17-oxosteroids are separated on a column of silica gel containing 25% (w/w) of water and 1·75% of methanol by the gradient elution technique with a mixture of water-saturated light petroleum and dichloromethane. The steroids in the separated fractions are identified by paper chromatography, and determined colorimetrically by the M.R.C. method (*Lancet*, 1951, **261**, 585). The effects of varying the composition of the stationary phase are reported, and the role of water in the separation is discussed. W. T. CARTER

2989. **The acid-catalysed solvolysis of dehydroepiandrosterone sulphate and its significance in the examination of urinary 17-oxosteroids. [Determination of dehydroepiandrosterone sulphate.]** L. Segal, B. Segal and W. R. Nes (Dept. of Chem., Clark Univ., Worcester, Mass., U.S.A.). *J. Biol. Chem.*, 1960, **235** (11), 3108–3111.—A 24-hr. sample of urine is neutralised with 5% aq. Na_2CO_3. One-third of the sample is made 3M with respect to NaCl and is extracted once with ethyl acetate (2 vol.); the organic phase is dried with anhyd. Na_2SO_4 (6 g per 100 ml of soln.). The dried and filtered extract is treated with sufficient H_2SO_4 in ethyl acetate to make the final concn. 2×10^{-4}M in H_2SO_4. The soln. is kept at 30° for 16 to 20 hr., then extracted once with N-NaOH (100 ml) and twice with water (100 ml). The organic layer is dried with anhyd. Na_2SO_4 and evaporated *in vacuo* at <40°. The residue is transferred with benzene (total vol. 4 ml) to a column (9 mm diam.) of Al_2O_3 (6 g) de-activated with water (4%). The column is eluted with a mixture of ethanol and benzene, starting with 0·2% and ending with 2% of ethanol in benzene. The dehydroepiandrosterone (androst-5-en-17-one) was determined in the eluates by the Zimmermann reaction. J. N. ASHLEY

2990. **Spectrophotometric determination of selenium in steroids.** L. J. Throop (Mead Johnson and Co., Evansville, Ind., U.S.A.). *Anal. Chem.*, 1960, **32** (13), 1807–1809.—The Se is separated from the organic compound with Raney nickel, oxidised to H_2SeO_3 with HNO_3, then converted with *o*-phenylenediamine into 1,2,3-benzoselenodiazole, the extinction of which is measured at 330 mμ. *Procedure*—Place the sample (containing 20 to 100 μg of Se) in a flask, dissolve it in 50 ml of ethanol and add about 300 mg of fresh Raney nickel prepared by the method of Drake (*Org. Synth.*, 1941, **21**, 15). Heat the soln. under reflux for 30 min., cool and decant the ethanol from the nickel, which is then washed by decantation with 30 ml of ethanol. Fit an air condenser and add 4 ml of HNO_3 dropwise with care. Warm the soln. to dissolve the nickel, cool and add 10 ml of H_2O. Boil the soln., cool and add 20 ml of H_2O and 10 mg of *o*-phenylenediamine dihydrochloride. Set aside for 15 min., then adjust the pH to 2·5 $\pm$ 0·1 with aq. NH_3. Transfer to a separating-funnel and extract with $CHCl_3$ (2×10 ml). Dry the $CHCl_3$ extract with anhyd. Na_2SO_4 and dilute to 25 ml with $CHCl_3$. Measure the extinction at 330 mμ against a $CHCl_3$ blank. Run a blank determination on the Raney nickel and subtract the value from the sample reading; Se (μg) = (extinction $\times$ 0·432 $\times$ 1·1)/(791 $\times$ 4). D. C. ARMSWORTH

2991. **Micro-method for amylase determinations using 0·05 ml of blood.** I. T. Beck, E. Pinter, R. D. McKenna and H. Griff (McGill Univ., Montreal, Canada). *Canad. Med. Ass. J.*, 1960, **82** (3), 152–153.—A modified Somogyi method is described. The sample of blood (0·05 ml) is added to 0·95 ml of 4% Na citrate soln. and is centrifuged, and 0·2 ml of the supernatant liquid is withdrawn for the amylase determination. The remaining 0·8 ml is treated with 1 ml of pptg. reagent (40 ml of water, 5 ml of 10% sodium molybdate soln. and 5 ml of 0·67N-H_2SO_4) and centrifuged for 5 min.; 1 ml of the supernatant liquid is used for the determination

of original blood-sugar by the Folin - Wu method. For the amylase determination, 3 tubes are prepared, *viz*, (*A*) a blank containing 1 ml of water, 0·4 ml of acid NaCl soln. (1% aq. NaCl containing 3 ml of N-HCl per litre) and 0·2 ml of pptg. reagent, (*B*) containing 1 ml of 0·5% starch soln., 0·4 ml of acid NaCl soln. and 0·2 ml of water, and (*C*) containing 1 ml of starch soln., 0·4 ml of acid NaCl soln. and 0·2 ml of the supernatant liquid from the blood. The tubes are incubated for 1 hr. at 37°, then treated with 0·5 ml of 7% $CuSO_4.5H_2O$ soln. and 0·5 ml of 10% sodium tungstate soln. and centrifuged for 10 min. Glucose is determined in the supernatant liquids (1 ml) by the Folin - Wu method. Amylase is calculated in Somogyi units from the weight of glucose formed after subtracting the blank. R. E. E.

2992. **Determination of amylase in enzyme preparations of animal origin.** K. Zipf and A. Schmid (Inst. für Pharmakol., Toxikol. und Pharm., Tierärtzl. Fak., Univ. München). *Arch. Pharm., Berlin*, 1960, **293** (10), 890–895.—Amylase is determined by degradation of a starch soln. and photometric evaluation of the remaining starch by reaction with iodine. Beer's law is obeyed for concn. of starch up to 20 mg. *Procedure*—Triturate a 500-mg sample with sand, extract with 50 ml of buffer soln. (pH 6·8) [4·64 g of KH_2PO_4 and 5·82 g of $Na_2HPO_4.2H_2O$ made up to 1 litre, and with 2% (v/v) of glycerol added] and dilute with buffer soln. to 1 litre. Centrifuge and filter. Dilute the filtrate so that 10 ml degrades $\geq$4 mg of starch per min. Heat 10 ml with 10 ml of starch mucilage (1 g of starch and 0·5 g of NaCl diluted to 500 ml) to 37°, mix, heat for 1 min., then add 2 ml of N-HCl and 10 ml of iodine reagent (1 g of iodine and 3 g of KI made up to 500 ml). Dilute an aliquot with 9 vol. of water at 37°, measure the extinction at 565 mμ after 10 min. against a blank, and determine the amylase by reference to a standard curve. The reproducibility of the method (15 determinations) is within ±0·9%. A. G. Cooper

2993. **Method for the direct detection of proteolytic enzymes after electrophoresis in agar gel.** J. Uriel (Service de Chimie Microbienne, Inst. Pasteur, Paris). *Nature*, 1960, **188**, 853–854.—The enzyme preparation is subjected to electrophoresis in the usual way, and then the agar plate is immersed in a bath containing a protein which is susceptible to degradation by the proteolytic enzyme. The plate is incubated, then fixed, dried and coloured by the method of Uriel and Grabar (*Anal. Abstr.*, 1957, **4**, 1000). The sites on the plate having a proteolytic activity towards the chosen substrate show up as weakly or non-coloured zones on an evenly coloured background. For each enzyme it is necessary to adjust the reaction conditions, namely the pH, the temp., the time of incubation, the substrate concentration, and the quantity of enzyme used. The proteolytic activity of trypsin preparations and an aq. extract of rabbit spleen has been studied (conditions given) by the method. The advantage of the method is that it can be seen whether the heterogeneity of enzyme preparations is due to the presence of several constituents possessing a proteolytic activity, or to the presence of non-enzymatically active protein impurities. R. A. Hendey

2994. **Simple method for determining δ-aminolaevulic acid dehydrase in blood and liver.** E. Grogg (F. Hoffmann-La Roche & Co., A.-G., Basel, Switzerland). *Bull. Schweiz. Akad. Med. Wiss.*, 1960, **16** (4), 305–308 (in German).—The method is based on the formation of porphyrins when δ-aminolaevulic acid (100 μg) is incubated with haemolysed erythrocytes or ground rabbit liver at pH 7·4 and 38° for 24 hr. After acidifying with acetic acid, the porphyrins are extracted with ether, re-extracted with 10% HCl and determined fluorimetrically against standard haematoporphyrin soln. E. C. Butterworth

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See also Abstracts—**2782**, Hg in biol. materials. **2840**, Cr in seaweed. **2997**, Flavonols in drugs. **3056**, Cyanamide in biol. fluids. **3070**, Chromatography of polynucleotides and proteins. **3077**, U.v. photography of spots on chromatograms of biological extracts. **3121**, Blood-gas analyser.

Pharmaceutical analysis

2995. **Table for the identification of pharmaceuticals by means of the Kofler heating block.** M. Brandstätter-Kuhnert and G. Kuhnert (Pharmakognost. Inst., Univ., Innsbruck, Austria). *Sci. Pharm.*, 1960, **28** (4), 287–303.—The m.p. determined on the Kofler heating bed differ from the values given by conventional methods for compounds that are liable to decompose, those that contain water of crystallisation and those that exist in unstable or metastable modifications. These differences are discussed and a table of values for ≃260 compounds is presented. S. M. Marsh

2996. **Detection of organic substances. [III.]** L. Rosenthaler (Seftigenstrasse 40, Bern, Switzerland). *Pharm. Acta Helv.*, 1960, **35** (12), 679–686 (in German).—The following are considered—colour reactions of alcohols and ketones, unsaturated compounds, aliphatic amines, aliphatic amino-acids, and substances with active methylene groups; reactions of vitamin K, ketones, morpholine and purines; tests to distinguish methanol from ethanol, quinoline from isoquinoline, and theobromine from theophylline; and the oxidation of alcohols with chloric acid. (*Cf. Anal. Abstr.*, 1961, **8**, 1188.) A. R. Rogers

2997. **Determination of the content of flavonol derivatives in drugs.** B. Christ and K. H. Müller (Pharm.-Wissenschaft. Abt. II, A. Nattermann & Cie, Köln-Braunsfeld, Germany). *Arch. Pharm., Berlin*, 1960, **293** (12), 1033–1042.—Mix the powdered drug (0·2 to 1 g) with acetone (20 ml) and 25% HCl (2 ml). If leucoanthocyanins are present, add 0·5% aq. hexamine soln. (1 ml). Heat under reflux for 30 min., and filter through cotton wool. Extract the residue repeatedly with boiling acetone, filter and dilute the combined filtrates to 100 ml with acetone. Dilute a 20-ml aliquot with H_2O (20 ml) and extract with ethyl acetate (15, 10, 10 and 10 ml). Wash the combined extracts with H_2O (2 × 40 ml) and dilute to 50 ml with ethyl acetate. Treat 10 ml with 0·5% aq. $NaNO_2$ soln. (0·5 ml) and a 2% soln. of $AlCl_3.6H_2O$ in 5% methanolic acetic acid (2 ml), dilute to 25 ml with 5% methanolic acetic acid, set aside for 45 min. and measure the extinction at 425 mμ against a reagent blank. A. R. Rogers

2998. **Thin-film chromatography in the analysis of drugs.** H. Gänshirt and A. Malzacher (Wissenschaftl. Abt. der Firma Dr. Karl Thomae G.m.b.H., Biberach an der Risz, Germany). *Arch. Pharm.*,

Berlin, 1960, **293** (10), 925–932.—Samples are chromatographed on films of Kieselgel G (Merck) containing 2% of Leuchtstoff ZS-Super (Riedel de Haen). The films show a green fluorescence at 254 mμ. Extraction procedures, eluents, and detection by fluorescence and by colour tests are described for caffeine (**I**), phenacetin (**II**) and acetylsalicylic acid in tablets; **I**, **II** and Valmorin [2-(2-chloroethyl)-2,3-dihydro-1,3-benzoxazin-4-one (**III**) in tablets; theophylline, papaverine and phenobarbitone in suppositories; **I**, **III** and phenylbutazone in tablets; amidopyrine, 5-ethyl-5-(2,5-endomethylene-1,2,5,6-tetrahydrophenyl)barbituric acid, phenylbutazone and codeine phosphate in suppositories; 2-butoxyethyl nicotinate, nicotinic acid and "nonoic acid vanillylamide" in ointments and liniments; and bisacodyl and its degradation products in suppositories and dragées.

A. G. COOPER

2999. **Rapid spectrophotometric determination of khellin, phenobarbitone, papaverine and theobromine in mixtures.** G. Machek (Biochemie-G.m.b.H., Kundl, Tyrol, Austria). *Sci. Pharm.*, 1960, **28** (4), 252–261.—The method depends on the differences in solubility of the four compounds. A weighed sample is extracted with 0·5N-NaOH and the extract is examined in a spectrophotometer at 275 mμ and 240 mμ against water. The two extinction values give the amounts of theobromine and phenobarbitone by reference to calibration curves. A second sample is extracted with 0·1N-HCl and the extract is examined at 251 mμ against a blank containing the requisite amount of theobromine and phenobarbitone. The extinction value is used to calculate the amount of papaverine present. A third weighed sample is extracted with hot ethanol and the extract is examined at 332 mμ against a blank containing papaverine. The value obtained gives the amount of khellin. The time required is 2 hr. The errors obtained were generally $< \pm 1\%$ for each constituent.

S. M. MARSH

3000. **Rapid electrophoresis of alkaloids.** L. A. Williams, Y. M. Brusock and B. Zak (Wayne State Univ. Coll. of Med., Detroit, Mich.). *Anal. Chem.*, 1960, **32** (13), 1883–1885.—A technique is described for preparing sheets of buffered agar gel with a parchment-paper backing on 3·25 in. × 4 in. glass plates. Alkaloids were separated from tissue extracts by electrophoresis in the gel at a max. of 350 V and 100 mA in 25 min. After drying, the sheet was stripped from the glass and the colour developed with iodoplatinic acid soln. The purple spots were cut out and shaken with 5 ml of a 1% Na_2SO_3 soln. in half-satd. $Na_2B_4O_7$ soln. at pH 12 to 13. The soln. was extracted with CCl_4. The aq. phase was again extracted with CCl_4 - isobutyl alcohol at pH 8·5 to 9 and the combined organic phases were extracted with 0·1N-H_2SO_4 (1 ml). The alkaloid was identified and determined by spectrophotometry of the aq. phase.

G. BURGER

3001. **Analysis of crude drug preparations. I. Paper chromatography of nux vomica extract, nux vomica extract powder and nux vomica tincture.** Takeshi Asada and Akira Okamoto (Welfare Pension Hosp., Kamifukushima, Fukushima-ku, Osaka). *Arch. Pract. Pharm., Japan*, 1959, **19** (2), 130–134.—A semi-circular filter-paper was used for the separation of alkaloids in crude drug preparations. Butanol - 30% acetic acid - water (2:1:1) gave the best separations.

II. Determination of strychnine and brucine in nux vomica preparations. Takeshi Asada and Akira Okamoto. *Ibid.*, 1959, **19** (4), 262–264.—The method was applied to the determination of strychnine (**I**) and brucine (**II**), butanol satd. with 10% aq. NH_3 being used as the developing solvent. The extinctions of the two fractions were determined at the absorption maxima of **I** and **II** (252 mμ and 302 mμ, respectively).

S. NATORI

3002. **Influence of the temperature of drying on the alkaloid content in ergot.** F. Kaczmarek and H. Speichert (Inst. of Medicinal Plants, Poznań, Poland). *Biul. Inst. Roślin Leczn.*, 1960, **6** (2), 147–154.—Seventeen methods of drying ergot were tested experimentally, and drying at 60° for 3·5 hr. was found to be the best. Drying at 18° to 22° gives good results, but requires about 70 hr. A loss in alkaloid content was observed when temp. >60° were used, and drying at 100° for 1 hr. resulted in a loss of about 14%.

B. K.

3003. **Absorption spectra of cardiac glycosides and aglycones in sulphuric acid.** B. T. Brown and S. E. Wright (Dept. of Pharmacy, Univ. of Sydney, Australia). *J. Amer. Pharm. Ass., Sci. Ed.*, 1960, **49** (12), 777–779.—The absorption spectra of 38 cardiac glycosides, aglycones and aglycone derivatives in 98% H_2SO_4 have been determined from 210 to 600 mμ, and wavelengths and $E^{1\%}_{1cm}$ values at maxima and minima are tabulated. The A-series compounds may be distinguished by maxima at 350 and 415 mμ; the B series by strong maxima at 490 and 530 mμ; and the C series by maxima at 320 and 390 mμ.

A. R. ROGERS

3004. **Studies on digitalis glycosides. VIII. Quantitative determination of digitalis glycosides in micro quantities and variation of glycoside content in the leaves of *Digitalis purpurea* L. according to season and growth-stage.** Daisuke Satoh, Takayuki Wada and Susumu Yamamoto (Res. Lab., Shionogi & Co., Ltd., Amagasaki, Osaka). *Ann. Rep. Shionogi Res. Lab.*, 1959, **9**, 21–32.—Digitalis glycosides are separated by paper chromatography with n-butanol - isoamyl alcohol - formamide - water (4:1:1:4). The digitoxin fraction is determined by the xanthhydrol method (Pesez, *Ann. Pharm. Franç.*, 1952, **10**, 104) and the gitoxin and strospeside fraction by the fluorimetric H_3PO_4 method (Pesez, *Brit. Abstr. C*, 1951, 224). The coeff. of variation is $\simeq 5\%$. The method is applied to the study of the seasonal variation of the glycosides.

S. NATORI

3005. **Application of the induced sodium azide - iodine reaction in quantitative analysis. V. Determination of microgram amounts of penicillin.** Z. Kurzawa and A. Suszka (Dept. of Gen. Chem., Polytech., Poznań, Poland). *Chem. Anal., Warsaw*, 1960, **5** (6), 897–902.—The method is based on the reaction induced by penicillamine, after the hydrolysis of penicillin. *Procedure*—The soln. of penicillin in H_2O (5 ml) is hydrolysed with N-NaOH (5 ml) for 30 min., neutralised with N-HCl (5 ml) and acidified with 0·1N-HCl (11 ml); 20% NaN_3 soln. (10 ml), 0·02N-iodine (10 ml) and H_2O to 100 ml are added with stirring. The soln. is titrated with 0·02N-Na_3AsO_3, with starch as indicator. The result is referred to a standard curve prepared by the same procedure with standard penicillin soln. The method can be used to determine 0·001 to 0·030 mg of penicillin or equivalent amounts of potassium or procaine penicillin. The accuracy is $\pm 5\%$.

B. K.

3006. **New chromatographic and colorimetric method for the determination of aloin.** B. Borkowski, M. Henneberg and I. Urszulak (Inst. of Medicinal Plants, Poznań, Poland). *Biul. Inst. Roślin Leczn.*, 1960, **6** (2), 125–135.—Dissolve 0·5 g of powdered aloin in 25 ml of methanol and apply 5 spots of this soln. (0·03 to 0·05 ml ≡ 0·6 to 1·0 mg of sample) to Whatman No. 1 paper. Develop the chromatogram for 18 to 20 hr., with the descending technique and Partridge's soln. [n-butanol - glacial acetic acid - H_2O (4:1:5)]. Dry the paper first at 20° and then for 5 min. at 70°, and locate the red spots of aloin (R_F 0·65 to 0·67) under u.v. light. Cut out the spots and extract the aloin by shaking with 5 ml of methanol for 30 min. Add 1 ml of piperidine and measure the extinction in a Pulfrich photometer, with a S42 filter. Compare with a standard curve. The method gives good results; the standard deviation is from 0·18% to 0·44%. B. K.

3007. **Tentative and rigorous proof in the identification of organic compounds, and application of these concepts to the detection of the active principles of marihuana.** N. D. Cheronis (Dept. of Chem., Brooklyn College, Brooklyn, New York). *Microchem. J.*, 1960, **4** (4), 555–567.—A discussion of the basic principles involved in the identification of organic compounds is followed by a review of methods used for the detection of the active principles of marihuana (*Cannabis sativa*).
A. J. Bennett

3008. **Colorimetric determination of glycyrrhetinic acid in liquorice root and its galenical preparations.** C. H. Brieskorn and G. H. Mahran (Inst. f. Pharm. u. Lebensmitt., Univ., Würzburg, Germany). *Arch. Pharm., Berlin*, 1960, **293** (12), 1075–1078.—Heat the powdered sample (100 mg) on a boiling-water bath for 2 hr. with conc. HCl (10 ml), cool, shake with ethyl ether and decant the ethereal layer from the brown resin at the interface. Dissolve the resin in 10% NaOH soln., mix with the acid layer and extract with more ether. Wash the combined ether extracts with H_2O (2 × 10 ml), shake with active carbon (0·1 g), filter and dilute to 50 ml with ethyl ether. Evaporate a 20-ml aliquot, containing <1·6 mg of glycyrrhetinic acid, dissolve the residue in 96% ethanol (1 ml) and add a 1% ethanolic soln. of 2,6-di-t-butyl-*p*-cresol (0·5 ml) and 20% NaOH soln. (1 ml). Heat on a boiling-water bath for 30 min., dissolve the residue in neutral acetone (20 ml) and measure the extinction at 575 mμ against a reagent blank. Subtract a correction for the colour due to reaction of ether-soluble triterpenes. A. R. Rogers

3009. **New colour reaction for the identification of sulphonamides.** V. Stefanović (Inst. Tuberculosis, Beograd, Yugoslavia). *Acta Pharm. Jugoslav.*, 1959, **9**, 151.—The sulphonamide (10 mg) is treated with 1 ml of 3N-H_2SO_4 and 0·1 ml of 10% vanillin soln. in 50% ethanol. A yellow to orange - red colour is produced. This reaction can be used for sulphanilamide, sulphadiazine, sulphaguanidine, sulphamethazine, sulphathiazole and sulphacetamide. Chem. Abstr.

3010. **Determination of phthalylsulphacetamide, chloroquine and di-iodohydrcxyquinoline in a mixture.** S. Bhattacharya and S. K. Ganguly (Bengal Immunity Res. Inst., Calcutta). *J. Instn Chem., India*, 1960, **32** (5), 225–227.—Phthalylsulphacetamide was extracted from the mixture with 10% $NaHCO_3$ soln. and the neutralised soln. was titrated with 0·05M-$NaNO_2$, with an external indicator of starch - iodide paste. Chloroquine phosphate was extracted with water, aq. NH_3 was added, the soln. was extracted with ethyl ether and the extract evaporated to dryness to obtain the chloroquine. Di-iodohydroxyquinoline was assayed in the mixture by the U.S.P. method.
S. A. Brooks

3011. **Analysis of phenothiazine derivatives employed in pharmacy. I.** F. Eiden and H.-D. Stachel (Pharm.-Chem. Inst., Univ., Marburg/Lahn, Germany). *Dtsch. ApothZtg*, 1960, **100**, 1369–1371.—Phenothiazine derivatives are identified by means of their u.v. spectra. Absorption max. in methanol soln. of the hydrochlorides of chlorpromazine, diethazine and ethopropazine, methotrimeprazine maleate, prochlorperazine dimaleate, promethazine hydrochloride, perphenazine phosphate, thioridazine hydrochloride and thiazinamium methyl sulphate are tabulated.
A. G. Cooper

3012. **Analytical and technical pharmaceutical investigations on chlorothiazide.** A. Baruffini and A. Tiengo (Ist. Chim. Pharm., Univ. Pavia, Italy). *Farmaco, Ed. Prat.*, 1960, **15** (11), 669–675.—Chlorothiazide (**I**) is accurately determined spectrophotometrically at concn. of ≃10 μg per ml in 0·1N-NaOH at 292 mμ if the soln. are fresh. In old soln., **I** is hydrolysed to 6-amino-4-chlorobenzene-1,3-disulphonamide (**II**), which interferes. Polarography at 25°, at a sensitivity of 1 to 2·51 × 10^{-7} amp. per mm, of 1·3 mM soln. in McIlvaine's buffer (pH 7) containing 0·004% of gelatin, gave a well-defined wave at −1·42 V, with height proportional to the concn. The reduction product is dihydrochlorothiazide, which gives no wave. The polarographic method is applicable in the presence of **II**. J. I. M. Jones

3013. **Determination of prednisolone in the presence of hydroxyzine and in formulations containing analgesics.** J. D. Duerr and B. A. Pappas (Chas. Pfizer & Co. Inc., Process Development Lab., Pharm. Prod. and Packaging Dept., Brooklyn, N.Y.). *J. Amer. Pharm. Ass., Sci. Ed.*, 1960, **49** (12), 759–761.—Carefully add conc. H_2SO_4 (5 ml) to an ethanolic extract of the sample (5 ml, containing ≃0·5 mg of prednisolone) in a 25-ml flask. Stopper and invert the flask, but do not shake it. Set aside for 5 min., add a 10% aq. soln. of $FeCl_3.6H_2O$ (0·02 ml), mix and set aside in a water bath at 20° for 5 min. Add ethanol (5 ml), shake, set aside at 20° for 5 min. and measure the extinction at 525 mμ against a reagent blank. Treat similarly a standard soln. of prednisolone. Hydroxyzine, aspirin, caffeine and phenacetin do not interfere. A. R. Rogers

3014. **New approach to the determination of vitamin A in pharmaceutical products.** R. Tardif (Res. Lab., Frank W. Horner Ltd., P.O. Box 959, Montreal, Canada). *J. Amer. Pharm. Ass., Sci. Ed.*, 1960, **49** (12), 741–745.—The vitamin A is preferentially extracted from the unsaponifiable matter with purified hexane. The soln. is examined spectrophotometrically at one wavelength (325 mμ) before and after treatment with H_2SO_4 to destroy the vitamin. Experiments with five multivitamin preparations and a sample of cod-liver oil indicate 100% recovery, in comparison with 82 to 96% by the method of the U.S.P. XV. The standard

deviation by the proposed method is smaller by a factor of three. The single-extraction method of Napoli *et al.* (*Anal. Abstr.*, 1960, **7**, 3522) is recommended for routine control determinations.

A. R. ROGERS

3015. **The limit test for sulphate ions in pharmacopoeial preparations.** H. Wojahn and E. Boll (Bundesgesundheitsamt, Max-von-Pettenkofer Inst., Berlin-Dahlem). *Dtsch. ApothZtg*, 1960, **100** (48), 1395–1398.—A procedure which involves pptn. with seeding with $BaSO_4$ in the presence of ethanol (*cf.* Zimmermann, *Anal. Abstr.*, 1956, **3**, 3450), as described in the Netherlands Pharmacopoeia, is recommended for inclusion in the D.A.B.VII. The results are more reproducible than those obtained by the D.A.B.VI method. A. R. ROGERS

3016. **Determination of calcium ions as impurities in pharmacopoeial preparations.** H. Wojahn and E. Boll (Bundesgesundheitsamt, Max von Pettenkofer Inst., Berlin-Dahlem). *Dtsch. ApothZtg*, 1960, **100** (50), 1453–1455.—Reproducibility of the Ca oxalate nephelometric procedure for the determination of Ca^{2+} present as impurities (Addendum 3 to D.A.B. VI) is poor, and the time required for complete pptn. varies according to the salt examined. Better reproducibility is attained by means of the inoculation crystal method of Krogh-Svendsen and Reimers (*Anal. Abstr.*, 1957, **4**, 1664), but results are often low. Satisfactory recoveries are obtained by addition of ethanol to the test and standard soln. before adding the inoculation reagent.

A. G. COOPER

See also Abstracts—**2968**, Oscillopolarography of barbiturates. **2969**, Chloramphenicol in body fluids. **2981**, Assay of L-threonine. **2992**, Amylase in enzyme preparations. **3052**, Disinfectant activity of quaternary ammonium compounds.

Food

Foods and food additives, beverages, edible oils and fats, vitamins.

3017. **A routine titrimetric method for the determination of invert sugars in refined white sugars.** J. Knight and C. H. Allen (Tate & Lyle Ltd., Plaistow Wharf Refinery, London, E.16). *Int. Sugar J.*, 1960, **16**, 344–346.—A method with a range adequate for refined sugar that is not very sensitive to time and temp. variations has been developed from that devised by Street (*Analyst*, 1958, **83**, 628). *Reagent*—Add 40 ml of N-NaOH to 600 ml of water. Add Na_2CO_3 (25 g) and Na K tartrate (25 g) and shake until dissolved. Add 6 g of $CuSO_4.5H_2O$ in 100 ml of water, and make up to 1 litre. *Procedure*—Dissolve 5 g of sugar in 5 ml of water, add 2 ml of alkaline copper soln. and shake. Immerse in a boiling-water bath for 5 min. and cool. Transfer to a dish and titrate with 0·005N-EDTA (disodium salt) to murexide (screened with methylene blue). I. DICKINSON

3018. **Analysis of endrin residues in flour by infra-red spectroscopy.** A. N. Bates (Agric. Res. Council, Pest Infestation Lab., Slough, Bucks., England). *J. Sci. Food Agric.*, 1960, **11** (12), 750–753.—The method depends on measurement of the extinction at 11·73 μ of a soln. of a purified extract of the sample. A light petroleum - acetone extract of the sample is purified by phasic separation with water and with 5% Na_2SO_4 soln. at 2°, followed by passage through a column of activated alumina, and elution with light petroleum. The eluate is evaporated and the residue is dissolved in iso-octane for i.r. spectroscopy, with a path-length of 2 mm. The extinction of the peak at 11·73 μ is measured from a base-line constructed between 11·45 and 12·30 μ, and the endrin content of the soln. is calculated from a standard curve. Recoveries of added endrin from white flour and bran at the 200 p.p.m. and 80 p.p.m. levels were from 78 to 106%. E. C. APLING

3019. **The role of amyl alcohol in the determination of fat by the Gerber method.** H. Hänni (Eidg. milchwirtschaftl. Versuchsanstalt, Liebefeld-Bern, Switzerland). *Milt. Lebensmitt. Hyg., Bern.*, 1960, **51** (6), 515–524.—It was found that differences between results by the Gerber method and those by the Röse - Gottlieb or Schmid - Bondzynski methods can be traced to the use of impure amyl alcohol. The suitability of a batch of amyl alcohol can readily be established by means of gas chromatography. A method is described.

I. DICKINSON

3020. **Rapid determination of volatile fatty acids in milk by circular paper chromatography and a modified colouring technique.** G. Hadland and O. Johnsen (Dairy Res. Inst., Vollebekk, Norway). *Int. Dairy Congr., Proc., 15th Congr., London*, 1959, **3**, 1539–1544.—A procedure is described for circular paper chromatography of volatile acids in milk and other substances. A series of 6 to 8 samples are run on a paper. Running time in the developing solvent (butanol, aq. NH_3 and H_2O) is 2·5 hr. After drying for 30 min. the paper is sprayed with a soln. of methyl red and bromothymol blue in ethanol and formaldehyde adjusted with NaOH to a pH of ≃11·5. The acids appear as clear red bands on a green background.

CHEM. ABSTR.

3021. **New procedure for the routine determination of the various non-casein proteins of milk.** R. Schaffenburg and J. Drewry (Nat. Inst. Res. Dairying, Shinfield, Reading, England). *Int. Dairy Congr., Proc., 15th Congr., London*, 1959, **3**, 1631–1637.—A quant. procedure was developed for the chemical partition of the non-casein proteins of milk by means of fractionation controlled by paper electrophoresis. The procedure consists of the determination of N on filtrates prepared with Na_2SO_4 at controlled pH. It subdivides the non-casein proteins into 4 fractions, *viz*, β-lactoglobulin, residual albumins (largely α-lactalbumin), globulins, and proteose peptones. These results are in substantial agreement with those obtained by U-tube electrophoresis. The method is simple, relying on determinations of the N content of easily prepared filtrates, and is suitable for routine analysis.

CHEM. ABSTR.

3022. **Determination of water in milk powder by drying in vacuum over phosphoric oxide as a reference method.** J. Eisses (Govt. Dairy Sta., Leyden, Netherlands). *Int. Dairy Congr., Proc., 15th Congr., London*, 1959, **3**, 1583–1588.—For the determination of the total H_2O content (including hydrate H_2O) of milk powder by drying *in vacuo* over P_2O_5, the drying temperature must be at least 90°. A satisfactory procedure for normal milk powder appeared to be drying at 90° for an initial period of 1·5 hr. followed by periods of 1 hr. until

the wt. is constant. Comparison of the results for normal milk powders with those obtained by other standard methods showed that results by the Karl Fischer and Dutch methods were approx. 0·1%, those by the British method 0·4%, and those by the German method 0·6% lower. CHEM. ABSTR.

3023. **Chromatographic method of detecting foreign fats in butter fat.** W. A. McGugan (Dairy Technol. Inst., Dept. Agric., Ottawa, Canada). *Int. Dairy Congr., Proc., 15th Congr., London*, 1959, **3**, 1534–1538.—The unsaponifiable material is chromatographed on a glass plate coated with a mixture of plaster of Paris and silicic acid. The plate is spotted with 25 μl of a soln. of the unsaponifiable matter from 2·5 g of oil in 25 ml of hexane. After the solvent has evaporated, the plate is placed vertically in a soln. of 5% ethyl acetate in hexane until the solvent front has risen 16 to 19 cm; the solvent is evaporated and the plate is sprayed with 10% HNO_3 in conc. H_2SO_4. The plate is then placed on an asbestos mat on a hot-plate and the spots are charred, becoming black or gray. Adulterated samples show spots which do not normally occur in butter fat. Most adulterants could be detected at the 10% level, but coconut oil, in 20% concn., could not always be detected. CHEM. ABSTR.

3024. **Paper-chromato-oscillopolarographic determination of vanillin and ethylvanillin in foods.** H. Woggon and U. Köhler (Inst. für Ernährung, Potsdam-Rehbrücke). *Ernährungsforschung*, 1960, **5** (4), 402–409.—In the method described, the two aldehydes in a suitable extract are first separated chromatographically and each is then determined by means of an oscillopolarograph. For vanilla sugar, 5 g of well-mixed sample is digested with 10 ml of aldehyde-free ethanol for 30 min. at 45° in a closed flask, with frequent shaking. A suitable amount of the cooled product is applied to Whatman 3 MM paper and developed with isobutyl alcohol - aq. NH_3 (5:3) as ascending mobile phase. The dried spots are located under u.v. light, and cut out and eluted with a total of 25 ml of 0·5M-semicarbazide hydrochloride (0·1N in HCl). The eluate (10 ml) is polarographed, the depth of the incision on the $dV/dt = f'(V)$ curve being measured and compared with a calibration curve based on a 3:1 mixture of vanillin and ethylvanillin. Starch-containing materials require a short preliminary treatment with conc. NaOH soln., followed by acidification and immediate extraction with ether, the ether being then replaced by ethanol. Tests of the method on model mixtures and commercial products gave reproducible results, an error of $\pm 10\%$ being found on 2 μg of vanillin or ethylvanillin per ml. The method is simple and rapid, and hence suitable for routine determinations.
C. L. HINTON

3025. **Analysis of food flavours by gas - liquid chromatography. Separation and identification of the neutral components from bread pre-ferment liquid.** D. E. Smith and J. R. Coffman (Central Res. Lab., General Mills Inc., Minneapolis 13, Minn.). *Anal. Chem.*, 1960, **32** (13), 1733–1737.—Ether-soluble neutral components in a bread pre-ferment liquid were separated and isolated on a 28% Ucon 50 HB-5100 column at 150°. Identification of the components was further carried out by i.r. spectroscopy, mass spectrometry and the preparation of derivatives. The 27 compounds detected included aldehydes, ketones, alcohols, esters and diols, many already known as fermentation products. Compounds not previously reported as fermentation products included ethyl formate, γ-butyrolactone, 3-hydroxypropyl acetate and 2-phenylethanol. C. B. BAINES

3026. **Specific colorimetric method for the determination of sorbic acid [in foodstuffs].** H. Schmidt (Bundesforschungsanstalt für Lebensmittelfrischhaltung, Karlsruhe, Germany). *Z. anal. Chem.*, 1960, **178** (3), 173–184 (in German).—Methods for the determination of sorbic acid (**I**) are reviewed. In the present method, **I** is isolated by steam-distillation; oxidation to malonaldehyde and reaction with thiobarbituric acid (**II**) gives a red colour. Other common preservatives do not interfere. *Procedure*—Weigh 1 to 5 g of sample (0·5 to 1 mg of **I**) into a flask, add enough H_2SO_4 to give 5 to 10 ml of soln. N in H_2SO_4, and 5 to 10 g of $MgSO_4.7H_2O$. Steam-distil for 10 to 15 min. and dilute the distillate to 250 ml. Transfer 2 ml to a test-tube (35 cm × 1·3 cm), add a 1:1 mixture of 0·01N-$K_2Cr_2O_7$ and 0·3N-H_2SO_4 (2 ml) and heat at 100° for 5 min. Add a 0·5% soln. of **II** (2 ml) and heat again for 10 min. Cool rapidly and measure the colour at 532 mμ. For the qual. detection of **I**, the steam-distillation can be omitted. Results are given for margarine, mustard, orange juice and horseradish. P. D. PARR-RICHARD

3027. **Oscillopolarographic determination of contaminating substances in foodstuffs.** V. Jedlička and A. Pašek (Centr. Res. Inst. of Food Ind., Prague). *Chem. Zvesti*, 1960, **14** (11–12), 757–763.—The oscillopolarographic methods for the determination of As^{3+}, CN^-, CS_2, S^{2-} and dimercaprol are reviewed and new methods are suggested for the determination of CS_2, in 0·25M-triethanolamine with N-NaOH as electrolyte, suitable for samples containing 2 to 100 μg of CS_2 per ml in the presence of trichloroethylene; H_2S can be determined in the same electrolyte. For the determination of tetramethylthiuram disulphide (1 to 15 μg per ml), M-citric acid is recommended as supporting electrolyte.
J. ZÝKA

3028. **Detection of mono- and poly-phosphates in "phosphate-free" materials used for the prevention of blood clotting and in the manufacture of sausages.** F. Ruf, K. Glandorf and O. Gehrig (Joh. A. Benkiser G.m.b.H., Chem. Fabr., Ludwigshafen am Rhein, Germany). *Mitt. Lebensmitt. Hyg., Bern*, 1960, **51** (6), 533–539.—A paper-chromatographic procedure and a gravimetric and a colorimetric method are described that are suitable for use in the meat industry; 5 mg of P_2O_5 per 100 g can be detected.
I. DICKINSON

3029. **Polarographic determination of the furfuraldehyde-producing compounds in beer.** J. Hummel (Forschungsanstalt für Brauerei und Mälzerei, Prague). *Brauwissenschaft*, 1960, **13** (10), 318–320.—Such compounds are determined by hydrolysing the beer with 12% HCl in a boiling-water bath for 6 hr., neutralising with aq. NH_3 and analysing the resulting filtrate polarographically with LiCl. Calibration curves are prepared with hydrolysates of arabinose. The content of furfuraldehyde-producing compounds is lower in beers of low nitrogen content. J. V. RUSSO

3030. **Determination of lactic acid [in wine] with ion-exchange resins.** V. Dimotaki-Kourakou (Lab. of the Wine Inst., Athens). *Ann. Falsif.*, 1960, **53**,

569–580.—The sample is treated with basic Pb acetate to precipitate polyhydric phenols and hydroxy-acids, and the filtrate is passed through a cation-exchange column (Dowex 50) to remove excess of Pb and other cations, and then through an anion-exchange column (Dowex 2) to adsorb lactic acid (**I**). The **I** is eluted with ammonium carbonate soln. and oxidised with ceric sulphate to acetaldehyde, which is titrated iodimetrically. For 10 synthetic samples containing up to 2 g of **I** per litre, the mean error was ±1·1%. G. RUSSELL

3031. **Chromatography of lipids on glass-fibre paper.** M. Černíková and J. Horáček (Masaryk Univ., Brno, Czechoslovakia). *Arch. klin. exp. Derm.*, 1960, **209**, 572–577.—The factors involved in the use of glass-fibre filter-paper for chromatographic separation of mixtures of fats were investigated. The paper was impregnated with either silica gel (Dieckert and Reiser, *J. Amer. Oil Chem. Soc.*, 1956, **33**, 123) or with water glass. For development of the chromatogram, 0·05, 2·0 and 5·0% soln. of ethyl ether in light petroleum (boiling-range 40° to 60°) were employed. Test substances examined were 1-monostearin, 1,3-distearin, cetyl alcohol, oleic acid, triolein, cholesterol, cholesteryl oleate, squalene, lipids of the blood serum of healthy humans, and lipids of heart muscle and of subcutaneous fatty tissues. Spraying with 50% H_2SO_4 and heating the paper to between 300° and 400° carbonised the fats and gave dark-brown spots; under these conditions cholesterol-containing lipids appeared as red spots. Before the treatment with acid and heat, phospholipids were determined with molybdic acid, and the NH_2 groups detected with ninhydrin. The basic requirement for satisfactory separation of the fats was a thorough saturation of the closed system and the glass-fibre paper with the solvent mixture. CHEM. ABSTR.

3032. **Gas chromatography for the determination of fatty acids in natural fats.** A. L. Prabucki (Inst. f. Tierernährung, Eidg. Tech. Hochschule, Zürich, Switzerland). *Mitt. Lebensmitt. Hyg., Bern*, 1960, **51** (6), 509–514.—The extraction, hydrolysis, esterification and gas-chromatographic separation of the monocarboxylic acid methyl esters are described, and the interpretation of the chromatogram is discussed. I. DICKINSON

3033. **Gas chromatography in the manufacture of stearin.** R. Aeschbacher (Seifenfabrik F. Steinfels A.-G., Zürich, Switzerland). *Mitt. Lebensmitt. Hyg., Bern*, 1960, **51** (6), 525–532.—The composition of the raw material and intermediate and end products was studied by gas chromatography. The apparatus and method are described and results are given. It was found that the iodine values and acid values calculated from the chromatographic results agreed well with those obtained by the usual chemical methods. I. DICKINSON

3034. **Spectrophotometric determination of sterculic acid [in maize oil].** A. J. Deutschman, jun., and I. S. Klaus (Dept. of Agric. Biochem., Univ. of Arizona, Tucson). *Anal. Chem.*, 1960, **32** (13), 1809–1810.—A modification of the Halphen test for the cyclopropene structure has been developed by which sterculic acid in maize oil in concn. between 1 and 7×10^{-4} g per g can be determined to within 10%. The oil (1 ml) is heated with CS_2 satd. with S (5 ml) and pyridine (5 ml) at 48° for 1 hr. and 108° for 45 min. After 2 hr. at room temp. the extinction is measured at 505 mμ. G. BURGER

3035. **Determination of diene conjugation for the evaluation of antioxidants.** Chotaro Tsukamoto, Shoji Ozeki and Kengo Yanase (School of Pharm., City Univ., Mizuho-ku, Nagoya). *Arch. Pract. Pharm., Japan*, 1959, **19** (3), 202–206.—The use of the u.v. absorption at 234 mμ for the determination of conjugated dienes, intermediates in the autoxidation of unsaturated fatty acids and fats, is proposed. The test sample is added to linseed oil and the mixture is heated on a water bath in a current of O. The u.v. absorption of the mixture is observed and the activity of the sample is deduced from the formation of diene compounds. The results agree fairly well with those by peroxide titration and by viscosity measurements. S. NATORI

3036. **Determination of vitamins. I. Vitamin D.** H. Janecke and I. Maass-Goebels (Inst. für Lebensmittelchem., Univ. Frankfurt, Germany). *Z. anal. Chem.*, 1960, **178** (3), 161–172 (in German).—Structural changes in vitamin D due to methods commonly used for its isolation, such as saponification and column chromatography, have been examined by u.v. spectrophotometry, and paper and thin-layer chromatography. A list of products and wavelengths of maximum absorption is given. It is concluded that choice of adsorbent is important and that saponification should be avoided if possible. The determination of small amounts of vitamin D is discussed in the light of the results obtained. P. D. PARR-RICHARD

3037. **Microbiological assay of thiamine. Two methods modified for use with small amounts of test material.** P. M. Scholes (Bact. Dept., Queen Elizabeth Coll., Univ. of London). *Analyst*, 1960, **85**, 883–889.—The methods modified are that of Fitzgerald and Hughes (*Ibid.*, 1949, **74**, 340) with *Lactobacillus fermenti* and that of Deibel *et al.* (*J. Bact.*, 1957, **74**, 818) with *Lb. viridescens*, and 2 mμg of thiamine can be determined. The preparation of cultures and inocula is described and the test substances used were dried yeast and the faeces and caecal contents of rats on experimental diets. For dried yeast, digestion and extraction procedures were those of Fitzgerald and Hughes (*loc. cit.*), adjusted for small amounts, one extract being made for the determination of thiamine and another in which the thiamine had been destroyed with sulphite. Rat faeces and caecal contents (for free thiamine only) were extracted with 0·1N-H_2SO_4 at room temp. The assays were made in E.E.L. colorimeter tubes with a pattern similar to that of Fitzgerald and Hughes (*loc. cit.*). The inoculated tubes were incubated for 17 hr. at 37° for *Lb. fermenti* and at 30° for *Lb. viridescens*, and the turbidities, read with an E.E.L. photo-electric colorimeter, were referred to standard graphs. Results are discussed, and the suggestions of Deibel *et al.* (*loc. cit.*) that *Lb. viridescens* is less susceptible than *Lb. fermenti* to inhibitory or stimulatory substances, and that it may provide the more convenient method, are supported. A. O. JONES

3038. **Effect of methanol on the conversion of thiamine to thiochrome.** B. S. Wostmann and P. L. Knight (Lobund, Dept. of Biol., Univ. of Notre Dame, Indiana). *Experientia*, 1960, **16** (11), 500–501 (in English).—Methanol (up to 3 ml) was included in the alkaline $K_3Fe(CN)_6$ reagent mixture (total vol. 8 ml) for the oxidation of thiamine to thiochrome. It was found that the inclusion of

methanol improved the conversion from ≃60% to 90%. The optimum amount of methanol appeared to be 2 ml. D. E. EVANS

3039. **Quantitative determination of ascorbic acid in heat-treated substances.** K. Szöke (Inst. f. Ernährungswissenschaft, Budapest). *Nahrung*, 1960, **4** (9), 825–831.—It is shown that the osazone produced by the reaction of ascorbic acid with 2,4-dinitrophenylhydrazine can be separated from interfering substances by filtration followed by paper chromatography. The osazone is determined by measuring the area of the spot, or by eluting and measuring the extinction of the eluate. Tomato purée was used as test material. I. DICKINSON

3040. **Reductone interference in the assay of ascorbic acid. A comparative study of different methods of analysis.** T. Kuusi (State Inst. for Tech. Res., Lab. for Food Res. and Technol., Helsinki, Finland). *Suomen Kem.*, 1960, **33** (7–8), 139–143 (in English).—The method of Robinson and Stotz with the formaldehyde condensation modification (*J. Biol. Chem.*, 1945, **160**, 217) seems to be the most reliable for the determination of ascorbic acid and reductones in rose-hip preparations. The paper-chromatographic method (*cf.* Heimann *et al.*, *Anal. Abstr.*, 1954, **1**, 369) may also be used for the quant. determination of ascorbic acid and qualitatively to check for substances that interfere with the ascorbic acid determination, but it is not suitable for the quant. determination of reductones. A. R. ROGERS

See also Abstracts—**2913**, Gas chromatography of food flavours. **2914**, Gas chromatography of fatty acids. **2955**, Oils, fats and waxes in cotton. **3014**, Vitamin A in pharm. products. **3061**, Malathion residues in cereals. **3075**, Determination of methyl and propyl *p*-hydroxybenzoates. **3084**, Gas-chromatographic separation of lipids.

Sanitation

Analysis of air, water, sewage, industrial wastes, industrial poisons.

3041. **Determination of small quantities of ozone in room air. Supplementary communication.** G. A. Hunold and W. Pietrulla (Bundesgesundheitsamt, Lab. f. chem. Toxikol., Berlin-Dahlem). *Z. anal. Chem.*, 1961, **178** (4), 271–274 (in German).—The method of Thorp (*Ind. Eng. Chem., Anal. Ed.*, 1940, **12**, 209), in which 33% KI soln. at pH 4·4 is used for absorbing O_3, is very sensitive to light. A simplified method of Byers and Saltzman (*Amer. Ind. Hyg. Ass. J.*, 1958, **19**, 251) is now described, in which 1% of KI in neutral phosphate buffer (0·1M-KH_2PO_4 - 0·1M-Na_2HPO_4) is used. Dil. H_2SO_4 is added to the soln. containing the liberated iodine, and the iodine is titrated with 0·005N-$Na_2S_2O_3$. Blanks are run with O_3-free air; the determination of 0·1 p.p.m. of O_3 takes 2·5 hr. (*Cf. Anal. Abstr.*, 1959, **6**, 3788.) B. B. BAUMINGER

3042. **Determination of sulphur dioxide in contaminated air. Modification of the iodimetric methods of Marston and Wells, of Thomas, and of Ellis.** O. Ramírez Torres (Dept. de Invest. Ind., Admin. Fomento Econ., San Juan, Puerto Rico). *Rev. Soc. Quím. Méx.*, 1960, **4** (6), 201–206.—The construction and use of a specially designed apparatus, equipped throughout with ground-glass joints, for the iodimetric determination of SO_2 in air are described. The apparatus provides for measurement of the vol. of air passed by means of a flow-meter and a chronometer, and for additions by micro-burette of 0·005N-iodine to the absorbing soln. (40 ml containing 2 g of starch and 1 g of KI per litre), without interrupting the air flow. Lower concn. of iodine (down to 0·00013N) are used in the absorbing soln. than in the method of Thomas and Cross (*Ind. Eng. Chem.*, 1928, **20**, 645), thus eliminating loss of iodine by volatilisation. E. C. APLING

3043. **Micro-determination of chloride in water.** W. Kemula, A. Hulanicki and A. Janowski (Dept. of Inorg. Chem., Univ., Warsaw, Poland). *Talanta*, 1960, **7** (1–2), 65–69 (in English).—The effect of pH on the Cl^- - Hg - diphenylcarbazone system is reported. For optimum results the pH should be maintained at 3·4 ± 0·1. Copper, Ni, Co, Zn, Cd and Pb interfere, and should be removed, *e.g.*, by extraction of their diphenylcarbazone complexes with $CHCl_3$ before the addition of Hg^{2+}. Errors on 10 to 50 μg of Cl^- are within ±1 μg. T. R. ANDREW

3044. **Flame-photometric determination of calcium and strontium in natural water.** Yasumitsu Uzumasa, Yoshikazu Nasu and Toshiko Seo (Fac. of Sci., Hokkaido Univ., Sapporo). *J. Chem. Soc. Japan, Pure Chem. Sect.*, 1960, **81** (3), 430–434.—Strontium is co-pptd. with Ca as oxalate, which is converted into oxide and then into chloride and examined flame-photometrically. The presence of Mg does not cause interference; Fe and Al are masked with Na citrate. The amount of Ca (0·5 to 1 g per litre) does not affect the intensity of the Sr lines. The working curve for Sr is linear for <20 mg of Sr per litre. Calcium can be simultaneously determined at 423 mμ, the background intensity at 440 mμ being deducted. Ethanol increases the sensitivity. *Procedure*—To the sample soln., containing 25 to 50 mg of Ca, add HCl (6N) (5 ml), Na citrate (1 g) and ammonium oxalate (3 g), warm, and neutralise with aq. NH_3 soln. (to methyl red). Set aside for 1 hr., filter, and ignite the residue. Dissolve the ash in water and HCl (6N) (2·5 ml) and make up to 50 ml after the addition of ethanol (5 ml). Measure the intensity of the Sr line at 461 mμ and that of the background at 470 mμ. K. SAITO

3045. **Analysis of natural waters with ion-exchange resin and a formate buffer as eluent.** Hiroyuki Tsubota (Nat. Inst. Radiological Sci., Kurosunacho, Chiba). *J. Chem. Soc. Japan, Pure Chem. Sect.*, 1960, **81** (6), 927–931.—An earlier method for separating alkaline-earth and alkali elements (Tsubota and Kitano, *Anal. Abstr.*, 1961, **8**, 444) was applied to rain and hot-spring water. From a column (*e.g.*, diam. 1·1 cm, length 10 cm) of Amberlite IR-120 (100 to 150 mesh), Na^+, K^+, Fe^{3+}, Al^{3+}, Mn^{2+}, Mg^{2+} and Ca^{2+} are eluted with formate buffers of increasing pH value (2·8 to 4·0) in the order given. For an analysis, Na^+ and K^+ are eluted with 0·2N- and 0·5N-HCl, respectively, and the other ions are eluted with the buffer soln. (30 to 50 ml each); Sr^{2+} and Ba^{2+} are then eluted with M- and 2M-ammonium formate, respectively. K. SAITO

3046. **Determination of strontium-90 and yttrium-90 in contaminated natural waters.** M. H. François and M. Jezequel (Service de Contrôle des Radiations et de Génie Radioactif, Fontenay-aux-Roses,

France). *Bull. Cent. Belge Étud. et Docum. Eaux*, 1960, No. 50, 269–271.—Total Y is extracted, after the addition of inactive Y as carrier, with 2-thenoyltrifluoroacetone (**I**) in benzene at pH 6·6, the extract is evaporated to dryness and its β-activity determined. Strontium remains in the aq. phase, and is extracted only at a pH of 8 to 9; Ra, U and Th do not form complexes with **I**, and Pu forms a complex only at high pH and when it is in the quadrivalent state. T. R. ANDREW

3047. **Determination of titanium in natural water.** J. Korkisch (Anal. Inst., Univ. Wien, Austria). *Z. anal. Chem.*, 1960, **178** (1), 39–42 (in German).—Titanium (5 to 100 μg) can be completely separated from commonly occurring elements such as Ca, Mg and Fe by adsorption as the ascorbate complex on to a column of Dowex 1-X8 (ascorbate form) (100 to 200 mesh). The ascorbates of V, W, U, Th and Zr are also adsorbed. The test soln., after filtration and addition of ascorbic acid, is adjusted to pH 4 to 4·5 and added to the column. The adsorbed Ti ascorbate complex is converted into the fluoride complex by the addition of 0·1N-H_2SO_4 containing NaF, and the Ti is then eluted with 0·1N-H_2SO_4 containing H_2O_2. The eluate is treated with ascorbic acid and the yellow colour formed at pH 3·8 is measured at 355 mμ. The detailed procedure is given.

B. B. BAUMINGER

3048. **Comparison of three methods of determining the concentration of uranium in sea water.** J. D. Wilson, R. K. Webster, G. W. C. Milner, G. A. Barnett and A. A. Smales (A.E.R.E., Harwell, Gt. Britain). *Anal. Chim. Acta*, 1960, **23** (6), 505–514 (in English).—An isotopic dilution method is described, involving the use of mass spectrometry and ^{235}U as tracer. In a fluorimetric method (*cf.* Milner and Barnett, *Anal. Abstr.*, 1959, **6**, 1127) ^{237}U is used as tracer. In both methods the U is extracted into $CHCl_3$ as its 8-hydroxyquinoline complex. Results obtained by the two methods and by a pulse polarographic method (*cf.* Milner *et al.*, A.E.R.E. Report R3227, 1960) are compared for precision, accuracy and time required. They give a mean value of 3·33 $\pm$ 0·08 μg of U per litre of water from the English Channel and the Bay of Biscay. The precision increases in the order fluorimetry, pulse polarography and isotopic dilution.

H. N. S.

3049. **Determination of urea in swimming-bath water.** J. Fuchs. *ChemikerZtg*, 1960, **84** (24), 805–806.—The xanthhydrol method gives lower results than the hypochlorite method in which intermediate products of the reaction between urea and HOCl also react and are calculated as urea. The hypochlorite method is therefore more suitable for determining the degree of contamination of chlorinated waters. *Procedure*—To 500 ml of water sample add 0·1N-Na_2CO_3 (30 ml), boil and filter. Neutralise the filtrate with 0·1N-HCl and add 5 ml in excess. Boil off CO_2, add 10% $BaCl_2$ soln. (5 ml) and filter. Add excess of $Ba(OH)_2$ soln. and filter off the ppt. formed on standing. Neutralise the filtrate with HCl, add 0·1N-NaOH (5 ml) and evaporate to 30 to 50 ml. Connect a NaOH-asbestos tube to the flask to avoid absorption of CO_2 during cooling. Add 0·1N-HCl (8 ml) and 200 ml of $Ba(OCl)_2$ soln. containing 3·5 to 6 g of HOCl per litre, then 25 mg of urea in aq. soln. After 10 min. add sufficient satd. $Ba(OH)_2$ soln. to produce a heavy ppt. Make just alkaline to phenolphthalein and shake. After 2 min. filter off the $BaCO_3$ ppt., wash it with hot water and 50% ethanol, then dissolve in 0·1N-HCl and back-titrate the soln. with 0·1N-NaOH; 1 ml of 0·1N-HCl $\equiv$ 3·004 mg of urea. The 25 mg of urea added is subtracted from the final result. A. J. BENNETT

3050. **Arsenic, germanium and selenium content in thermal waters in Misasa hot springs and a determination of phosphate ions in hot springs.** Masanobu Sakanoue (Balneological Lab., Okayama Univ., Misasa, Tottori-ken). *J. Chem. Soc. Japan, Pure Chem. Sect.*, 1960, **81** (2), 242–246.—In the presence of As^V (>100 μg per litre), the molybdenum blue method for phosphate gives high values. In such a case, the sample (almost neutral, 50 ml) is treated with HNO_3 (8N) (7·5 ml) and $(NH_4)_2MoO_4$ soln. (10%) (2 ml) and extracted with a mixture of $CHCl_3$ and n-butanol (3:1) (10 ml) after 15 min. The org. layer is shaken with $SnCl_2$ soln. (1% in 0·4N-HCl) (10 ml) and the extinction of the upper layer is measured at 745 mμ. K. SAITO

3051. **Application of gas chromatography to sludge digestion gas analysis.** W. N. Grune (Sch. of Civil Engng, Georgia Inst. of Tech., Atlanta, U.S.A.). *Wat. & Sewage Wks*, 1960, **107** (10), 396–399.—Gas-chromatographic methods for the detection and determination of H_2S, H and Ar have been examined. A number of partition columns have been used and the results are summarised.

C. A. SLATER

3052. **Method for laboratory evaluation of disinfectant activity of quaternary ammonium compounds.** British Standards Institution (2 Park Street, London, W.1). B.S. 3286:1960. 18 pp.—A suspension of viable organisms (*Escherichia coli*, *Pseudomonas aeruginosa* or *Staphylococcus aureus*) is added to soln. containing various concn. of the sample and the numbers of organisms surviving after a given time are determined.

See also Abstracts—**2822**, Thoron in air. **3102**, Determination of Be in air. **3120**, Methane in mine air. **3122**, ClO_3 in air.

Agricultural analysis

Soil, fertilisers, herbicides, pesticides, animal feeding-stuffs.

3053. **Estimation of potassium in soils by determination of potassium-40 content.** J. Rodriguez and G. E. Mattingly (Chem. Dept., Rothamsted Exp. Sta., Harpenden, Herts., England). *J. Sci. Food Agric.*, 1960, **11** (12), 717–721.—The potassium contents of 38 soils from Great Britain and Spain were determined by measuring their count rates in a cylindrical Geiger-Müller tube, 16 to 23 g of sample being used. A standard curve of counts per min. per g of K *vs.* density, prepared from measurements made on mixtures of powdered quartz and minerals of known potassium content and density, is reproduced. The potassium content of soils is calculated from the formula—K (%) = $110\ C/(W \times C')$, where C is the count rate of the sample, W is wt. (g) of sample, and C' = count rate per g of K at the apparent density of the sample. Results obtained on 26 soils from Central, Eastern and Southern England were on average 0·19% of K higher than by two methods of chemical analysis (gravimetric and flame photometric), but differences of up to +0·56% were

encountered with soils from Wales, Scotland and Spain. The standard error of the mean of duplicate determinations is ±0·075% of K for the radiochemical method compared with ±0·023 and ±0·026% of K for the two chemical methods. The radiochemical method is simple and requires no chemical treatment of the air-dried sample.

E. C. APLING

3054. **Fertiliser sampling to protect consumers and producers.** E. Fischer and H. Marr (Dept. Agric. Tech. Services, Pretoria, S. Africa). *J. Agric. Food Chem.*, 1960, **8** (5), 412–417.—The method of setting up acceptance sampling plans for fertilisers emanating from manufacturing units whose product is of constant quality is explained for any given levels of producer and consumer risks. Process variability is assumed to be known and constant; successive samples are taken at intervals sufficiently far apart in time for their properties to be uncorrelated. An acceptance procedure is also outlined for the case when several properties must simultaneously conform to specification.

B. J. WALBY

3055. **Joint Symposium on Fertiliser Analysis, London, April 1960.** Organised by The Fertiliser Society and The Society for Analytical Chemistry. Publ.: The Fertiliser Society, 44 Russell Sq., London, W.C.1. 215 pp.—The following papers are included. **The determination of molybdenum, iodine, fluorine, cobalt and copper,** R. F. Milton, pp. 21–27. **The determination of boron, magnesium, manganese and zinc,** S. M. Boden, pp. 27–35. **The determination of iron, chromium and nickel,** J. B. E. Patterson, pp. 35–38. **The determination of nitrogen,** H. N. Wilson, pp. 51–82. **The determination of potassium,** E. W. Schwehr and H. R. Conan, pp. 99–125. **The determination of phosphorus,** P. Craven and E. W. Schwehr, pp. 139–175. **The determination of the phosphorus content of rock phosphate,** H. L. Davies, J. Lacey, H. A. E. Mackenzie, A. Mendelowitz and J. Rijkheer, pp. 187–208.

3056. **Spectrophotometric determination of cyanamide.** D. A. Buyske and V. Downing (Lederle Lab., American Cyanamid Co., Pearl River, N.Y.). *Anal. Chem.*, 1960, **32** (13), 1798–1800.—To determine cyanamide (1 to 50 μg) in extracts from complex mixtures, the extract is adjusted to pH 7 and mixed with an aq. soln. of trisodium pentacyanoammineferrate buffered to pH 10·5. The extinction of the red complex is measured at 530 mμ. Beer's law is followed up to 52·5 μg of cyanamide. None of the compounds commonly found in cyanamide preparations interferes. Procedures are given for the determination of cyanamide in soil, blood and urine. The recovery is better than 93%.

G. BURGER

3057. **Determination of trichloroacetate when incorporated in an N.P.K. fertiliser.** I. Johnston (Levington Res. Sta., nr. Ipswich, Suffolk, England). *J. Sci. Food Agric.*, 1960, **11** (12), 695–700.—A column partition chromatographic method for the separation of acetic and mono-, di- and tri-chloroacetic acids and its application to the determination of about 0·5% of trichloroacetate in fertilisers are described. The chromatographic column (550 mm × 16 mm) is packed with Hyflo-Supercel supporting a stationary phase of 17N-H_2SO_4, and the eluting solvent is a mixture of dichloromethane and CCl_4 (1:1). The individual acids are detected and determined by titration of fractions of the eluate with N-Na methoxide. From fertilisers the free acid is extracted with ethyl ether from an aq. extract of the sample after acidification with H_3PO_4, the ether is evaporated at low temp., and the residue, mixed with anhyd. Na_2SO_4, is extracted with eluting solvent for application to the column. Recoveries of added trichloroacetate are 97 ± 2%.

E. C. APLING

3058. **Rapid determination of carotene in roughage and dried green feed by warm extraction with light petroleum.** K. Papendick (Staatl. Lehr- Versuchsanst. Grünlandwirtsch. Futterbau, Eichhof-Bad Hersfeld). *Landw. Forsch.*, 1960, **13**, 78–88.—A simplified form of a method previously published (*Ibid.*, 1955, **7**, 130; 1956, **8**, Sonderheft 7) was developed. From 2 to 10 g of air-dried green meal or hay (the latter chopped or ground to pass a 2-mm sieve and, if very damp, pre-dried with conc. H_2SO_4), is extracted for 2 hr. at 80° with 50 ml of light petroleum; the extract is cooled and 10-ml portions are chromatographed on 6 to 8-cm columns of activated alumina. Carotene (defined as carotenoids with vitamin-A activity) is eluted with light petroleum and determined colorimetrically (*e.g.*, with a Zeiss-Elko II colorimeter and filter S47 and a calibration curve, or from the extinction coeff. compared with that of a carotene standard). Results for samples of hay, lucerne hay and dried lucerne cubes agreed with those obtained by cold extraction with methanol and light petroleum. Four duplicate determinations can be made in 3·5 to 4 hr., or, with continuous working, 20 to 30 duplicate determinations daily. Owing to the short time taken, elaborate precautions against loss of carotene are not necessary. NUTR. ABSTR. REV.

3059. **The detection of organic phosphorus compounds.** J. C. Gage (Ind. Hygiene Res. Lab., I.C.I. Ltd., The Frythe, Welwyn, Herts., England). *Med., Sci. & Law*, 1961, **1** (2), 137–144.—A lecture.

3060. **Semi-quantitative paper-chromatographic determination of residues of systemic insecticides in plant materials.** J. Eichenberger and L. Gay (Chem. Lab. der Stadt, Zürich, Switzerland). *Mitt. Lebensmitt. Hyg., Bern*, 1960, **51** (6), 423–455.—Procedures for the extraction of the material, purification of the extract and transfer of the aliquots on to the paper, and the chromatographic technique used, are described in detail. Results are given. Residues from unknown insecticides can be identified by their R_F values.

I. DICKINSON

3061. **Recommended methods of analysis of pesticide residues in foodstuffs. Determination of malathion residues in cereals and oil-seeds.** Report by the Malathion Panel set up jointly by The Scientific Sub-Committee of the Inter-Departmental Advisory Committee on Poisonous Substances used in Agriculture and Food Storage, The Analytical Methods Committee of the Society for Analytical Chemistry and The Association of British Manufacturers of Agricultural Chemicals. *Analyst*, 1960, **85**, 915–921.—The sample (50 g of cereal or 25 g of ground oil-seed) is extracted with CCl_4 for 4 hr. in a Soxhlet extractor. To the extract, adjusted to 100 ml, are added 1 ml of a soln. of 1 ml of CS_2 in 200 ml of CCl_4 and 25 ml of ethanol and the mixture is shaken with 75 ml of Na_2SO_4 (anhyd.) soln. (9% w/v) acidified with 2·5 ml of HCl. The organic layer is filtered and shaken for exactly 1 min. with

25 ml of ethanol and 1 ml of 6N-NaOH and then for 1 min. with 75 ml of the Na_2SO_4 soln.; the CCl_4 layer is discarded, and the aq. layer is shaken for 30 sec. with 25 ml of CCl_4, which is again discarded. Another 25 ml of CCl_4 is added with phenolphthalein indicator and 6N-HCl until the pink colour is discharged and then 1 ml of $FeCl_3$ soln. (5 g of $FeCl_3.6H_2O$ in 100 ml of N-HCl). After shaking for 30 sec. the CCl_4 layer is discarded; the extraction is repeated twice, the CCl_4 layer being discarded each time. The aq. layer is shaken with 15 ml of CCl_4 and 1 ml of $CuSO_4$ soln. (3·5% w/v of $CuSO_4.5H_2O$), the separated CCl_4 layer is dried with anhyd. Na_2SO_4 and its extinction is measured at 418 mμ and referred to a calibration graph, which is linear in the range 50 to 250 μg of malathion.

A. O. JONES

3062. **Determination of *OO*-dimethyl *S*-(*N*-methylcarbamoylmethyl) phosphorothiolothionate [dimethoate] in technical Rogor and its formulations.** L. F. Dupée, K. Gardner and P. Newton (Fisons Pest Control Ltd., Cambridge, England). *Analyst*, 1960, **85**, 924.—The statement previously made (*Ibid.*, 1960, **85**, 177) that, in the determination of dimethoate by partition chromatography, *OO*-dimethyl phosphorodithioic acid (**I**) preceded dimethoate when eluted from the column with a mixture of isopropyl ether and light petroleum has been proved by subsequent work to be incorrect. The sample of this acid used to investigate its qual. behaviour was impure, and the peak formerly attributed to **I** resulted from an impurity (probably *OOS*-trimethyl phosphorodithioate). With the pure acid no significant acidic or phosphorus-containing material was eluted between 0 and 500 ml. Technical Rogor is unlikely to contain large amounts of **I**, and any traces present will be eluted long after dimethoate, so that they will interfere neither with the original determination nor with subsequent ones.

A. O. JONES

3063. **Biological methods for the analysis of technical Rogor, of formulations thereof and of residual insecticides on treated plants.** P. de Pietri-Tonelli and A. Barontini (Ist. di Ricerche Agrarie, Signa, Firenze, Italy). Publ.: Soc. Gen. per l'Ind. Min. e Chim., Milano. 1960. 12 pp.—Bioassays of Rogor [*OO*-dimethyl *S*-(*N*-methylcarbamoylmethyl) phosphorothiolothionate] were conducted with *Drosophila melanogaster* Meig (adult), *Culex pipiens* L. (larvae) or *Musca domestica* L. (adult). Full details are given.

J. I. M. JONES

See also Abstracts—**2782**, Hg in grain. **2827**, Determination of P_2O_5 in superphosphate. **2971**, Lichenin in oats. **2979**, Glutamic acid decarboxylase in wheat. **3018**, Endrin residues in flour.

5.—GENERAL TECHNIQUE AND APPARATUS

General

3064. **Abrasion of mortars of different composition.** N. W. H. Addink (Nat. Lab. N. V. Philips' Gloeilampenfabr., Eindhoven, Netherlands). *Chem. Weekbl.*, 1960, **56**, 622.—To ascertain the impurities that are introduced into a sample when it is powdered in a mortar, 1-g samples of quartz were powdered in mortars of different composition for 10 min. and the powders were analysed by emission spectrography. Mortars of agate, alumina and boron carbide (3 types) were investigated. The results are tabulated. It is recommended to clean a mortar by powdering a portion of the sample in it, rejecting the powder and then rubbing the mortar with dry cotton-wool or filter-paper; the mortar can then be used to powder the sample to be analysed.

M. J. MAURICE

3065. **Density hydrometers and specific gravity hydrometers.** British Standards Institution (2 Park Street, London, W.1). B.S. 718:1960. 34 pp.—The number of series of specified hydrometers has been reduced from nine to five. Each series covers the density (g per ml) or sp. gr. range 0·60 to 2·00.

3066. **Anti-foaming device for the Kjeldahl digestion.** G. Melnyk (Dept. of Agric., Cornell Univ., Ithaca, New York). *Chemist Analyst*, 1960, **49** (4), 118 (in English).—A hollow perforated polyethylene stopper is supported in the neck of the flask with copper wire. In cases of extreme foaming, two stoppers can be used with glass wool packed between them.

W. T. CARTER

3067. **Free-diffusion sample holder [for thermogravimetry].** P. D. Garn and J. E. Kessler (Bell Tel. Lab. Inc., Murray Hill, N.J.). *Anal. Chem.*, 1960, **32** (13), 1900–1901.—Certain advantages in restricting the decomposition products in thermogravimetric studies have been pointed out (*Anal. Chem.*, 1960, **32**, 1563). The advantages of the opposite extreme, in which the sample is heated in the form of a thin layer, are now described.

D. C. ARMSWORTH

3068. **Micro gas-chamber for the detection of volatile substances. Detection of fluoride.** F. L. Hahn (Escuela de Cienc. Quím., U.N.A.M., Mexico 12, D.F.). *Chemist Analyst*, 1960, **49** (4), 113 (in English).—The apparatus consists of a small Perspex block drilled to form a reaction chamber, with a connecting hole for the addition of reagents. The chamber is covered with a thin sheet of plastic which can be used to support a hanging drop of reagent or, when suitably perforated, for the support of a reagent paper.

W. T. CARTER

3069. **Acoustic method of gas analysis.** V. I. Daragan-Suschov and S. S. Anisimov (Central Sci. Res. Lab., Gosgortekhnadzora, RSFSR). *Zavod. Lab.*, 1960, **26** (12), 1368–1369.—An ultrasonic gas-analyser is described.

G. S. SMITH

Chromatography, ion exchange, electrophoresis

3070. **A modified calcium phosphate for column chromatography of polynucleotides and proteins.** R. K. Main, M. J. Wilkins and L. J. Cole (U.S. Naval Radiol. Defense Lab., San Francisco, Calif., U.S.A.). *J. Amer. Chem. Soc.*, 1959, **81**, 6490–6495.—Specimens of $Ca_3(PO_4)_2$, prepared at various pH values and subsequently modified by heat or alkali, were studied as possible adsorbents for the column chromatography of polynucleotides and proteins. One such specimen, pptd. at pH 6·7 and boiled with satd. $Ca(OH)_2$ soln., has the desired characteristics of ease and reproducibility of preparation, high flow rate, stability, capacity and degree of resolution. It was used to fractionate deoxyribonucleic acid and polyadenylic acid, to separate the latter from

the former, and to separate albumin from the nucleic acid of a 10:1 mixture of the two by gradient elution with neutral phosphate buffers. CHEM. ABSTR.

3071. **Chromatographic analysis with gradient elution.** P. Lebreton (Lab. de Chim. Biol., 93 rue Pasteur, Lyon, France). *Bull. Soc. Chim. France*, 1960, (11–12), 2188–2200.—A review is presented, with 70 references. It includes mathematical derivations of the laws governing elution, and their experimental verification. J. R. HUDSON

3072. **The theory of solid-phase diffusion in chromatography.** D. C. Bogue (Dept. of Chem. Engng, Univ. of Delaware, Newark, Del.). *Anal. Chem.*, 1960, **32** (13), 1777–1778.—It is shown that the linearised solid-phase mass-transfer equation which has been studied for frontal chromatography can also be applied to elution chromatography. G. BURGER

3073. **Discussion of thermometric monitor for chromatographic streams.** C. M. Proctor (2540 122nd Ave. S.E., Bellevue, Wash.). *Anal. Chem.*, 1960, **32** (13), 1897–1898.—Suggestions are made for the modification of the thermometric monitor circuit to avoid biasing the grid of the first section of the 12AY7 valve as suggested by Blumer (*Anal. Chem.*, 1960, **32**, 772). The bias resistor chain R_2 and R_3 can be omitted and the first grid connected directly to earth when that section of the valve is not in use. C. B. BAINES

3074. **Thin-layer chromatography.** H. Wagner (Univ., Basel, Switzerland). *Mitt. Lebensmitt. Hyg., Bern*, 1960, **51** (6), 416–423.—A lecture is presented in which various adsorption media and techniques are described. (36 references.) I. DICKINSON

3075. **Investigation of the quantitative evaluation of thin-layer chromatography. I. Ultra-violet spectroscopic determination of mixtures of methyl and propyl *p*-hydroxybenzoates by thin-layer chromatography.** H. Gänshirt and K. Morianz (Firma Dr. Karl Thomae G.m.b.H., Biberach-an-der-Riss, Germany). *Arch. Pharm., Berlin*, 1960, **293** (12), 1065–1075.—Separate 50-μg quantities of methyl *p*-hydroxybenzoate (**I**) and propyl *p*-hydroxybenzoate (**II**) on Kieselgel G (Merck) that has been purified by extraction with methanol. Use a mixture of pentane and acetic acid (22:3) as running solvent; elute with methanol and determine by spectrophotometry at 256 mμ. Correct for background absorption by a base-line method. Correct for irreversible adsorption on the gel. The coeff. of variation is estimated to be ±3·0% for **I** and ±3·8% for **II**. A. R. ROGERS

3076. **The use of ion-exchange resin-loaded paper for "desalting" small samples prior to paper chromatography.** R. M. Smillie (Biol. Dept., Brookhaven Nat. Lab., Upton, N.Y., U.S.A.). *J. Chromatography*, 1960, **4** (6), 494–495 (in English).—The sample is applied to the top of a strip of paper impregnated with cation-exchange resin (H^+ form) to the bottom of which is attached a similar strip impregnated with anion-exchange resin (OH^- form). The strips are eluted with a fixed amount of water. The ion-free eluate drips on to a sheet of chromatographic paper and the size of the spot is controlled by the use of a jet of air directed on to the underside of the paper. W. T. CARTER

3077. **Ultra-violet photography of fluorescent spots on chromatograms of biological extracts.** D. Abelson (Dept. of Int. Med., Yale Univ. School of Med., New Haven, Conn., U.S.A.). *Nature*, 1960, **188**, 850–851.—A simple technique is described for photographing chromatographic spots that fluoresce in u.v. light at 360 mμ. A Kodak 2A gelatin filter (enclosed in 0·0625-in. glass plates), which absorbs radiant energy below 405 mμ, is placed between the chromatogram and the contact paper ("Kodabromide F 5"). The chromatogram is exposed for ≃30 sec. to u.v. light from a 365-mμ source at a distance of 1 ft. The photograph is developed in the usual way. This technique dispenses with the need for a camera, and the information supplements that obtained from photographs of chromatographed compounds absorbing in the 254-mμ region. It is useful for substances such as 5-hydroxyanthranilic acid, which are not detected in 254-mμ photographs. R. A. HENDEY

3078. **Investigations by electron microscopy on the problem of chromatography on paper made from glass fibre.** G. Jayme, G. Hahn and H. Knolle (Inst. für Cellulosechem. mit Holzforschungsstelle der Tech. Hochsch., Darmstadt, Germany). *Z. anal. Chem.*, 1960, **178** (3), 194–198 (in German).—The separation of sugars on glass-fibre paper impregnated with (*a*) a phosphate buffer (0·1M-Na_2HPO_4 - H_3PO_4) (pH 5·0) (*cf.* Jayme and Knolle, *Angew. Chem.*, 1956, **68**, 243) or (*b*) SiO_2 pptd. *in situ* (*cf.* Dieckert and Reiser, *J. Amer. Oil Chem. Soc.*, 1956, **33**, 123) occurs by partition chromatography in the first case and by adsorption chromatography in the second. These differences in the nature of the paper have been confirmed by electron microscopy. (4 illustrations.) P. D. PARR-RICHARD

3079. **Preliminary recommendations on nomenclature and presentation of data in gas chromatography.** International Union of Pure and Applied Chemistry. *Pure and Appl. Chem.*, 1960, **1** (1), 177–186.—The recommendations include definitions relating to apparatus, retention parameters, reagents, chromatogram records and apparatus performance. G. J. HUNTER

3080. **Recent developments in gas - liquid chromatography.** A. T. James (Nat. Inst. for Med. Res., Mill Hill, London, N.W.7). *Lab. Practice*, 1960, **9** (12), 835–841.—A review, with 17 references.

3081. **Practical application of gas chromatography.** W. Simon (Org.-Chem. Lab., Eidg. Tech. Hochschule, Zürich, Switzerland). *Mitt. Lebensmitt. Hyg., Bern*, 1960, **51** (6), 470–509.—A literature review covering the various applications of gas chromatography. A list of commercially available apparatus is included. (104 references.) (*Cf. Chimia*, 1960, **14**, 189; *Anal. Abstr.*, 1961, **8**, 2.) I. DICKINSON

3082. **Construction of capillary columns of Pyrex glass for gas-phase chromatography.** A. Kreyenbuhl (Cerchar. Lab., Verneuil-en-Halatte, Oise, France). *Bull. Soc. Chim. France*, 1960, (11–12), 2125–2127.—Constructional details are given of an apparatus suitable for the manufacture of helical capillary columns from standard lengths of glass tube. Essentially there are four components arranged in series, *viz*, (*i*) a guide system and feed-motor, (*ii*) a straight furnace, (*iii*) a guide system and drawing-motor and (*iv*) a curved bending furnace. The feed-motor passes the glass tube into

the straight furnace at a rate of 4·6 mm per min., while the drawing-motor feeds capillary into the curved furnace at 340 mm per min. A tube 1·5 metres long yields 100 to 110 metres of capillary of diameter 0·62 ± 0·03 mm in the form of a helix of diameter 20 cm. J. R. HUDSON

3083. **Gas chromatography of high-boiling compounds on low-temperature columns.** C. Hishta, J. P. Messerly and R. F. Reschke (Bristol Lab., Syracuse, N.Y.). *Anal. Chem.*, 1960, **32** (13), 1730–1733.—By using glass beads as the solid support, adsorption on the column is minimised and reduction of the stationary phase to less than 0·3% is possible. This allows the rapid elution of high-boiling compounds at lowered temp. A comparison is made between Celite and glass-bead columns for polar and non-polar sample mixtures. Chromatograms from glass-bead columns are given which show good resolution and short retention times at temp. as much as 150° below the boiling-point of the sample. Results for a mixture of 7 ketones boiling from 57° to 156° and for a mixture of 3 hydrocarbons boiling from 174° to 253° are presented. C. B. BAINES

3084. **The use of silicone rubber gums or grease in low concentration as stationary phase for the high-temperature gas-chromatographic separation of lipids.** N. Nicolaides (Inst. of Marine Resources, Dept. of Food Sci. and Technol., Berkeley, Calif., U.S.A.). *J. Chromatography*, 1960, **4** (6), 496–499 (in English).—Retention times are given for high-molecular-weight hydrocarbons, esters and sterols on columns containing 5% (w/w) of silicone rubber gum or silicone grease on Chromosorb at 250° to 300°. W. T. CARTER

3085. **Micro-scale collection tube for gas chromatography.** H. J. Gold (U.S. Fruit and Vegetable Products Lab., U.S. Dept. of Agric., Winter Haven, Fla.). *Chemist Analyst*, 1960, **49** (4), 112 (in English).—A U-shaped collection tube is described and illustrated. A short vertical limb is sealed to the bottom of the U, and the condensed material is collected in this by centrifuging. W. T. CARTER

3086. **Detection methods and detectors used in gas chromatography.** M. Wurst (Res. Inst. Org. Synth., Pardubice-Rybitví, Czechoslovakia). *Chem. Listy*, 1960, **54** (10), 1042–1058.—The principles of detection measurements and the evaluation of detectors are reviewed. (52 references.) J. ZÝKA

3087. **An ultra-sensitive ionisation detector for permanent gas analysis.** R. Berry (U.K.A.E.A., Culcheth Lab., Warrington, Lancs.). *Nature*, 1960, **188**, 578–579.—A radioactive ionisation-type detector similar to the argon detector of Lovelock (*Anal. Abstr.*, 1959, **6**, 3297) was used with He as carrier gas. The metastable He atoms (19·8 eV) formed ionise all elements and compounds except Ne. The technique may therefore be used for the analysis of permanent gases whose ionisation potentials range from 12 to 16 eV. With a molecular sieve 5A column (4 mm × 30 cm) it is possible to determine $<0{\cdot}001$ µl of H, Ar, O, N, CH_4 or CO in < 2 min. The limit of detection is $\simeq 10^{-12}$ mole of component per ml of carrier gas. N. E.

3088. **High-temperature gas-liquid chromatography. Exploratory studies using an ionisation-detector chromatograph.** B. J. Gudzinowicz and W. R. Smith (Special Projects Dept., Monsanto Chemical Co., Everett, Mass.). *Anal. Chem.*, 1960, **32** (13), 1767–1771.—An instrument is described for the qual. and quant. determination of certain aliphatic and aromatic organic compounds having boiling-points >500°. A standard ionisation chromatograph is easily modified, and the accuracy and precision of this instrument with µg amounts of sample are comparable with those of conventional instruments. Special reference is made to quant. studies of isomeric terphenyls, with triphenylmethane as internal standard. C. B. BAINES

3089. **Ionisation detectors for gas chromatography.** P. H. Stirling and H. Ho (Canadian Industries Ltd., Toronto). *Ind. Eng. Chem.*, 1960, **52** (11), 61A–62A; 64A.—Ionisation detectors were found to give better sensitivity in determining separation by gas chromatography because they respond directly to the sample molecules in the column effluent and are thus relatively independent of the properties of the carrier gas. The most effective ionising process appeared to be collision of the sample molecules with metastable noble-gas atoms. Argon, hydrogen flame, gas discharge, high vacuum and β-ionisation detectors are described. NUCL. SCI. ABSTR.

3090. **Reduction of noise in thermal-conductivity detectors for gas chromatography.** R. Kieselbach (Engng Res. Lab., E. I. du Pont de Nemours & Co., Inc., Wilmington, Del.). *Anal. Chem.*, 1960, **32** (13), 1749–1754.—The technique described provides a 50-fold improvement in signal to noise ratio when using thermistor detectors. The major part of the noise in such detectors arises from sources external to the thermistor, and techniques for the elimination of noise due to convective heat transfer, ambient temperature changes and bridge power-supply variations are described. Circuit diagrams are given. C. B. BAINES

3091. **Use of a conventional mass spectrometer as a detector for gas chromatography.** L. P. Lindeman and J. L. Annis (California Res. Corp., Richmond). *Anal. Chem.*, 1960, **32** (13), 1742–1749.—The use of conventional magnetic-field mass spectrometers as auxiliary gas-chromatography detectors in a directly-coupled arrangement is described. This combination permits the simultaneous determination of several components in chromatographic peaks which contain more than one component, and of components appearing as shoulders on other peaks. Examples of the technique are given, including the analysis of a known mixture of 20 C_7 and C_8 saturated hydrocarbons and the analysis of a C_5 to C_8 California naphtha. C. B. BAINES

3092. **A two-recorder integrator-readout system for gas-liquid chromatography.** K. L. Jackson and C. Entenman (Biol. and Med. Sci. Div., U.S. Naval Radiological Defense Lab., San Francisco, Calif., U.S.A.). *J. Chromatography*, 1960, **4** (6), 435–445 (in English).—Two recorders fitted with ball and disc integrators receive the signal from the same detector. One recorder plots a predetermined fraction of the scale plotted by the other, giving a large effective expansion in scale and allowing a considerable increase in the sample size which can be analysed in a single run. The pulsed signals from the integrators are fed to register units coupled to rotary stepping relays, which may be operated manually or automatically as each peak is completed. The integrated peak areas agree with the results obtained by manual planimetry to within 1%. W. T. CARTER

3093. **Determination of the areas of resolved and partially resolved [gas] chromatography peaks.** J. C. Bartlet and D. M. Smith (Food & Drug Directorate, Dept. of National Health & Welfare, Ottawa, Canada). *Canad. J. Chem.*, 1960, **38** (11), 2057–2065.—Symmetrical chromatographic peaks are shown to correspond with Gaussian distribution curves, and their areas can be calculated in terms of the peak height and standard deviation (σ), both of which are obtained from measurements of the peak; σ is shown to vary linearly with retention time. The mathematical treatment is extended to the correction of the peak heights of incompletely resolved peaks. In these cases the area is calculated from values of σ derived from the retention times instead of from the peaks. Asymmetrical peaks, in which the skewness is $\pm$ 0·83, and overlapping peaks, one of which is asymmetrical, can be treated similarly. A. C. R. HARTLEY

3094. **Ion-exchange chromatography.** J. P. Redfern. *Lab. Practice*, 1960, **9** (12), 842–846.—A review, with 19 references.

3095. **Use of a diaphragm-type burette tap and a constant-level device for flow-rate control in ion-exchange chromatography.** R. A. Edge (Univ. of Cape Town, S. Africa). *J. Chromatography*, 1960, **4** (6), 501–502 (in English).—A flow rate of 0·5 ml per sq. cm per min., varying by $\simeq$5% over a 12-hr. period, can be obtained by fitting a plastic diaphragm-type burette-tap to the column exit and controlling the level of the solvent in the column by using as the reservoir a polyethylene bottle, the only outlet from which is immersed in the solvent at the top of the column. W. T. CARTER

Optical

3096. **Methods of evaluation of results in quantitative spectral analysis.** M. Malínek (Inst. of Metallurgy, Acad. Sci., Prague). *Chem. Listy*, 1960, **54** (11), 1144–1154.—A survey, with 33 references, is presented. J. ZÝKA

3097. **New method of quantitative spectrographic determination of elements.** V. V. Gusarskii and I. A. Kuzovlev. *Zavod. Lab.*, 1960, **26** (12), 1375–1378.—A method of calculation, in which the background intensity is automatically allowed for, is described. G. S. SMITH

3098. **Application of the digital computer to spectrochemical analysis.** J. N. Weber (Dept. of Geol., McMaster Univ., Hamilton, Ont., Canada). *Spectrochim. Acta*, 1960, **16** (11–12), 1435–1441 (in English).—Computer techniques are discussed for dealing with such problems as regression analysis, correlation analysis and the precision of measurement of spectral-line densities, and the calculation of concn. of a given element from photographic spectral-line densities. The simplicity of programming for contemporary computers is emphasised. E. G. CUMMINS

3099. **The Tesla spark source and its practical application in spectrographic analysis.** L. Paksy (Tech. Univ., Miskolc, Hungary). *Magyar Kém. Foly.*, 1960, **66** (12), 498–501.—Spectra of Al, Si, Fe, Mn, Cr, Ni and Zn obtained with Tesla and Feussner spark discharges are compared with reference to the variations in intensity of selected lines with position in the spark gap. Measurements are made on a Q24 spectrograph, the required portions of the spark being isolated by means of a diaphragm. Values for detection limits and reproducibility are tabulated. Calibration curves obtained with the Tesla spark discharge are also given, and it is claimed that the Tesla spark has some advantages for use in both qualitative and quantitative analysis. B. VASZKO

3100. **Spectrochemical analysis of solutions. A comparison of five techniques.** W. K. Baer and E. S. Hodge (Mellon Inst., Pittsburgh, Pa., U.S.A.). *Appl. Spectroscopy*, 1960, **14** (6), 141–146.—The porous cup, rotating disc, flat top, rotating platform and plastic cup techniques have been compared under three different excitation conditions and their applicability to practice is reviewed. The effects on the spectra of the inclusion of HCl, H_2SO_4, HNO_3 and H_3PO_4 are also discussed. E. G. CUMMINS

3101. **The double rotating spark-gap in spectral solution analysis.** W. Guttmann and G. Müller-Uri (Inst. f. Ernährung in Potsdam-Rehbrücke, Berlin, Germany). *Acta Chim. Acad. Sci. Hung.*, 1960, **25** (3), 327–340 (in German).—The double rotating spark-gap is described in detail and compared with the conventional spark-gap. The advantages of the method are discussed, particularly those arising from the constancy of the formation of the image of the spark in the prism. Parabolic electrode ends give more reproducible results than flat ones. The mean error in 40 soln., examined with a photoelectric cell, was 2%. R. PRESSER

3102. **Monitor for the quantitative determination of beryllium in the atmosphere.** M. S. W. Webb, R. J. Webb and P. C. Wildy (U.K.A.E.A., Research Group, Woolwich, London). *J. Sci. Instrum.*, 1960, **37** (12), 466–471.—Air is drawn through a triggered intermittent arc and the ratio of the line-to-background intensity of the 3130-A Be doublet is recorded on a ratio recorder. The instrument is calibrated by observing an auxiliary spark between beryllium - copper electrodes. The limit of detection is 1 μg per cu. metre for a 20-sec. sampling time. The performance is unaffected by particle size and by the chemical form of the beryllium. G. SKIRROW

3103. **X-ray fluorescence analysis of solutions.** M. M. Kakhana. *Zavod. Lab.*, 1960, **26** (12), 1359–1363.—A review with 26 references is presented. G. S. SMITH

3104. **Spectrochemical analysis using controlled atmospheres with a simple gas jet.** C. S. Annell and A. W. Helz (U.S. Geol. Surv., Washington, D.C.). *U.S. Geol. Surv. Profess. Paper*, No. 400–B, 1960, 497–499.—A controlled-atmosphere jet permits gas to surround the base of the electrode and to rise through the annular space between the electrode and the 0·5-in. opening of the ceramic "welding" tip: the top of the sample electrode projects about 5 mm above the rim of the jet. The best gas mixture was found to be Ar - O (4:1). The controlled atmosphere is effective in reducing the cyanogen band spectra; background corrections are also eliminated, thus enabling the working curves of affected spectral lines to be extended to a lower concn. range. R. A. HOWIE

3105. **Photomultiplier soft-X-ray spectrometer.** L. Jacob, R. Noble and H. Yee (Royal Coll. Sci. and Technol., Glasgow, Scotland). *J. Sci. Instrum.*, 1960, **37** (12), 460–463.—Constructional details are

given of a soft-X-ray spectrometer in which a beryllium - copper photomultiplier is mounted as a detector at the end of a radial arm pivoted at the centre of a Rowland circle. The instrument has been applied to the emission band-spectra of Al, Mg, Na and Li. G. SKIRROW

3106. **Semi-automatic plotting attachment for the Beckman DU spectrophotometer.** G. N. Bowers, jun., and S. Raymond (Hartford Hospital, Conn., U.S.A.). *Anal. Chem.*, 1960, **32** (13), 1901–1902.—A description is given of a typewriter attachment for eliminating the recording and manual plotting of extinction as a function of time. D. C. ARMSWORTH

3107. **Report on calibration of wavelength and photometric scales of non-recording spectrophotometers.** International Union of Pure and Applied Chemistry. Commission on Optical Data. *Pure and Appl. Chem.*, 1960, **1** (1), 147–162.—Nomenclature definitions are given. The wavelengths of spectral lines suitable for calibration purposes are tabulated. Topics discussed include the effects of stray light and the finite width of slits. The calibration of the photometric scale is considered in terms of the use of variable apertures, grids, rotating sectors, polarisation devices, the application of the inverse square law, chemical absorption methods and electrical methods. G. J. HUNTER

3108. **Modified test-tube holder for Beckman Model DU spectrophotometer.** G. C. Riggle (Div. of Res. Services, Nat. Inst. of Health, Bethesda, Md., U.S.A.). *Chemist Analyst*, 1960, **49** (4), 116 (in English).—The holder is modified to hold three test-tubes of different sizes simultaneously. Three measurements with different cell lengths can be made very rapidly without opening the instrument. W. T. CARTER

3109. **Lettering pen-holder for use with Beckman DK2 recording spectrophotometer.** I. Sunshine and L. Cordek (Western Reserve Univ., Cleveland, Ohio, U.S.A.). *Chemist Analyst*, 1960, **49** (4), 118 (in English).—A block-mounting for a reservoir lettering-pen is described. It has the advantage that pens can be interchanged without having to re-zero the spectrophotometer. W. T. CARTER

3110. **Temperature-control system for use with a spectrophotometer.** S. W. Thorne (Div. Plant Ind., C.S.I.R.O., Canberra, A.C.T.). *J. Sci. Instrum.*, 1960, **37** (12), 478–480.—Circuit details are given of a thermistor-controlled system for use with standard 5-cm cells. Temperatures in the range 30° to 80° can be selected with an accuracy of $\pm 0{\cdot}1°$ and with a control variation of about $\pm 0{\cdot}05°$. G. SKIRROW

3111. **Applications of complementary tristimulus colorimetry. I. Analysis of binary and ternary colorant systems.** H. Flaschka (School of Chem., Georgia Inst. of Technol., Atlanta, U.S.A.). *Talanta*, 1960, **7** (1–2), 90–106 (in English).—It is shown that, provided that the individual complementary colour co-ordinates (obtained by taking extinction values instead of the conventional transmission values) are known, mixtures of two or three colorants may be evaluated (by algebraic or graphical - algebraic methods) from measurements of the complementary colour co-ordinates of the system. The procedure is tedious, but results of high precision ($\pm 1\%$) are reported for mixtures of the complexes of Cu, Ni and Co with EDTA. T. R. ANDREW

3112. **Dilution - addition method for flame spectrophotometry.** F. S. Grimaldi (U.S. Geol. Surv., Washington, D.C.). *U.S. Geol. Surv., Profess. Paper*, No. 400–B, 1960, 494–495.—Matrix effects are largely overcome by sufficient dilution of the sample, residual matrix effects being corrected for by a "spiking" technique. The resulting plot of intensity *vs.* concn. defines the upper region of a working curve characteristic of the given sample matrix mixed with different amounts of test element. The curve is usually rectilinear, and its shape for lower concn. is deduced by extrapolation from the known portion to zero intensity, thus defining at the same time the point of zero concn. The extrapolation to zero intensity is simplified by choosing a concn. of standard such that, if the standard is set at 100, the unspiked sample reads in the range 20 to 35. Before plotting, the intensities are corrected for background by reading emissions on both sides of the line used. R. A. HOWIE

3113. **The effect of solution viscosity on the atomising process in flame-photometric analysis.** E. Pungor and M. Hangos (L. Eötvös Sci. Univ., Budapest). *Magyar Kém. Foly.*, 1960, **66** (12), 516–518.—The feeding of the soln. to the flame, either directly from the atomiser or via a mixing chamber, was studied. Soln. of LiCl (10^{-3}M) in ethanol - water mixtures of varied proportions were fed to the apparatus, and the speed of atomisation was measured as a function of ethanol concn., the air-pressure fed to the atomiser and the radius of the feed capillary. Certain of the results, which are depicted graphically, are compared with theoretical predictions based on the Hagen - Poiseuille law. Graphs showing the effect of the composition of the gas mixture fed to the atomiser on the intensity of the lithium emission are also given; a H - O - N flame was used. B. VASZKO

3114. **Effect of organic and aqueous solvents on flame-photometric emission and atomic absorption spectroscopy.** J. W. Robinson (Esso Research Lab., Baton Rouge, La., U.S.A.). *Anal. Chim. Acta*, 1960, **23** (5), 479–487 (in English).—In the flame photometry of metals the use of soln. in organic solvents, in place of aq. soln., enhances the emission signals. This enhancement is greater than can be accounted for by the higher flame temp. Neither can the higher temp. account for the enhancement of absorption when organic solvents are used in atomic absorption spectroscopy. Combustion mechanisms are suggested according to which organic solvents produce in the flame a greater concn. of the substance in the atomic condition, both in the excited state for greater emission and in the ground state for greater absorption. H. N. S.

3115. **Device for high-precision time-resolved spectroscopy.** Á. Bardócz (Acad. Sci., Central Res. Inst. Phys., Budapest, Hungary). *Spectrochim. Acta*, 1960, **16** (10), 1173–1175 (in English).—Details are given of a rotating-disc device which permits the selection of an arbitrary time range from the light emission of a transient discharge. The spark is initiated by light from a control lamp passing through an aperture in the slit and falling on to a photocell which actuates an electronic trigger. The device has been applied to the study of the spectrum in air of spectroscopically pure carbon electrodes, and it is shown that the air spectrum persists for only about 10 μsec. under the conditions employed. G. SKIRROW

3116. Purification of hydrocarbon solvents for spectrophotometry. G. D. Johnson (Kansas State Univ., Manhattan, U.S.A.). *Spectrochim. Acta*, 1960, **16** (11–12), 1489–1491.—The light transmission at < 250 mμ of the spectroscopic grades of iso-octane, heptane, cyclohexane and decalin has been improved by passage through Davison 12 activated silica gel. Other samples of silica gel and activated alumina tested had the opposite effect. E. G. CUMMINS

3117. Improvements in the potassium bromide disc technique. R. Kopff, R. Rueff and H. Benoît (Centre de Rech. sur les Macromolecules, Strasbourg, France). *Bull. Soc. Chim. France*, 1960, (10), 1694–1695.—The 12-mm disc press described is claimed to produce less cracking in discs than that of Franck (*cf. Z. Naturf.*, 1954, **9b**, 276). The spectra given illustrate the purification of KBr by the removal of H_2O and NO_3^-. Analytical-grade KBr (25 g) is dissolved in a mixture of twice-distilled H_2O (70 ml) and freshly distilled HBr (40%) (10 ml). Complete dissolution is achieved near the boiling-point. The material obtained by gently boiling the soln. to about half its volume and then cooling is recrystallised from twice-distilled H_2O and dried at a temp. near the m.p. for 30 min. The yield is 60 to 72%. E. G. CUMMINS

3118. Infra-red spectrophotometric analysis of phases adsorbed on powdered solids. M. Courtois and S. J. Teichner (Fac. Sci., Univ. Lyon, France). *Bull. Soc. Chim. France*, 1960, (10), 1773–1775.—The apparatus described has been designed to support a disc in an infra-red spectrophotometer under variable conditions of temp. and pressure. It consists of an evacuable cylindrical tube (measuring 110 mm $\times$ 35 mm), fitted with calcium fluoride windows, an internal heater and a cooling jacket. A pressure of 10^{-6} torr and a temp. of 100° can easily be obtained. Silica discs, prepared from particles 150 A in diam., have been used to obtain the spectra of $Ni(OH)_2$ adsorbed thereon.
E. G. CUMMINS

3119. Application of atomic absorption to chemical analysis. A review. D. J. David (Div. of Plant Industry, C.S.I.R.O., Canberra, A.C.T., Australia). *Analyst*, 1960, **85**, 779–791.—The theoretical basis of atomic absorption as far as it concerns analytical equipment is considered briefly. The instrumental arrangements are described, in particular, light sources and means of vaporisation of the sample, and the isolation, detection and evaluation of the lines. Atomic absorption is compared with other analytical methods and the conditions governing precision and accuracy are described. The sensitivity and precision of determinations of 20 metallic elements are tabulated. (45 references.)
A. O. JONES

3120. Interferometer. G. K. Subbotin and I. I. Zharikov. U.S.S.R. Pat. 131,969 (20.9.1960).—A safe interferometer for determining methane and CO_2 in mine air contains a chamber for the sample, through which light from a lamp is passed through a matt or opal-glass plate to obtain an even illumination, then through the gas chamber, and then totally reflected back through the chamber, and directed to an eyepiece, through which the interference pattern, which is independent of the position of the lamp, is examined. C. D. KOPKIN

Electrical

3121. Interference-free integration of a blood-gas analyser into a polygraphic system for recording physiological activities. J. B. Harlin and M. Cohen (Elect. Res. Dept., Lockheed Aircraft Corp., Burbank, Calif., U.S.A.). *Rev. Sci. Instrum.*, 1960, **31** (11), 1245–1246.—The use of a Beckman/Spinco model-160 gas analyser in conjunction with the multi-channel recorder Sanborn model (158–1100C) results in a 60 c/s modulation of the trace caused by the electro-mechanical chopper in the analyser. This interference also appears in other traces of the recorder owing to conductive coupling between the analyser and the other trace electrodes. These difficulties are overcome by feeding the analyser output into a recorder insensitive to 60 c/s chopper modulation. The recorder (Brown/Honeywell) is modified by the addition of a re-transmitting, slide-wire potentiometer, and the output from this is fed into the multi-channel recorder.
G. SKIRROW

3122. An instrument for recording the chlorine dioxide content of air. K. Schwabe, G. Fiehn and K. Spiegel. *Chem. Tech., Berlin*, 1960, **12** (12), 726–729.—The apparatus is designed for the continuous measurement of ClO_2 in hot (80°) water-saturated air. It functions by cathodic reduction of the ClO_2 dissolved in tap water. Under controlled conditions the current strength is proportional to the ClO_2 content of the gas.
N. E.

3123. New electrode system sensitive to very small gaseous samples. Takeru Higuchi and G. J. Papariello (School of Pharm., Univ. of Wisconsin, Madison, U.S.A.). *Talanta*, 1960, **7** (1–2), 127–128 (in English).—An electrode system is described consisting of a length of capillary tubing (Pyrex glass, Vycor or porcelain) heated to 500° to 700°, having an external spiral platinum cathode wrapped round it and an internal platinum anode, bent to make light frictional contact with the wall of the tube. At applied voltages greater than a critical value for each carrier gas (*e.g.*, 245 V for Ar and 450 V for N) the current passed by the system is markedly affected by the presence of traces of impurities in the gas stream. T. R. ANDREW

3124. Continuous polarographic analysis. IV. Carbon as measuring electrode. J. V. A. Novák (Polarographic Inst., Czech. Acad. Sci., Prague). *Coll. Czech. Chem. Commun.*, 1960, **25** (12), 3098–3104 (in German).—A description is given of polarographic instruments for the continuous measurement of the concn. of H_2S and SO_2 in technical gases. Although carbon electrodes cannot be used for the production of polarographic curves, they are very suitable for the continuous measurement of concn. at a constant potential, as no solid non-conducting film is formed on them. A carbon anode is used having a large effective area (2 to 3 sq. cm), which permits the use of robust measuring instruments. A copper cathode is used in the SO_2 instrument, but in the H_2S instrument a zinc amalgam cathode is necessary. The gas is bubbled through H_2SO_4 (0·05 to 1M), which is also flowing continuously through the electrode cell. The concn. measured range from 0·01 to 10% (v/v). The SO_2 instrument has been in continuous use for over 2 years without change of electrode.
A. J. BENNETT

3125. **Device for the synchronisation of two dropping-mercury electrodes.** W. H. Lockwood (U.K.A.E.A. Res. Group, Chem. Div., C.37 Royal Arsenal, Woolwich, London). A.E.R.E. Report R3521, 1960. 5 pp.—The design of a solenoid device for the simultaneous detachment of drops from two electrodes, suitable for use with the differential cathode-ray polarograph (*cf.* Davis and Seaborn, A.E.R.E. Report R3472), is discussed and the final design is described and illustrated. The essential features are an armature momentum of $\simeq$5000 g-cm per sec. and a lateral displacement of the capillaries of 0·05 mm. The solenoid is actuated by a pulse from the polarograph.

W. T. CARTER

3126. **Irreversible systems in stationary electrode polarography.** W. H. Reinmuth (Chem. Dept., Columbia Univ., New York, 27). *Anal. Chem.*, 1960, **32** (13), 1891–1892.—Potential - time relations in voltammetry with linearly changing potential and electrochemically irreversible reactions are useful in making qual. distinctions between reversible and irreversible systems, and permit the simple determination of kinetic parameters in irreversible cases. D. C. ARMSWORTH

3127. **Studies on alternating-current polarography. I. Modified instrument with a circuit for compensating current.** Masayoshi Ishibashi, Taitiro Fujinaga and Atsuyoshi Saito (Chem. Dept., Fac. Sci., Kyoto Univ., Sakyo-ku). *J. Chem. Soc. Japan, Pure Chem. Sect.*, 1960, **81** (5), 753–757.—Breyer's polarograph (*Aust. J. Sci. Res., A*, 1950, 567) was modified by attaching an auxiliary circuit consisting of a resistor and a capacitor, which compensates for the capacity and the resistance of the electrolytic cell. Both are changed step-wise with a rotary switch, so that no current is recorded in the absence of depolariser. The polarogram of Cd in HCl and in NH_4Cl is illustrated. K. SAITO

3128. **Phase-selective alternating-current polarography at stationary electrodes.** D. E. Smith and W. H. Reinmuth (Havemeyer Hall, Columbia Univ., New York, 27). *Anal. Chem.*, 1960, **32** (13), 1892–1893.—In the determination of trace amounts of metals by voltammetric methods, the limiting factor seems to be the capacitive current associated with the charging of the electrical double layer. An extension of an earlier technique (Adams *et al.*, *Ibid.*, 1960, **32**, 1526; Juliard, *J. Electroanal. Chem.*, 1959, **1**, 101; *Nature*, 1959, **183**, 1040), in which an electronic phase-sensitive detector permits the rejection of the charging current while retaining the major portion of the faradaic current, appears to have extremely high sensitivity.

D. C. ARMSWORTH

3129. **Progress in alternating-current oscillographic polarography.** R. Kalvoda (Polarographic Inst., Acad. Sci., Prague). *Chem. Listy*, 1960, **54** (12), 1265–1278.—A survey is presented of recent communications dealing with the theory, electrode processes, apparatus and use of $dE/dt = f(E)$ curves in qualitative and quantitative analysis. (31 references.) J. ZÝKA

3130. **Applications of oscillographic polarography in quantitative analysis. XII. Possible uses of the capacity effect.** R. Kalvoda (Polarographic Inst., Czech. Acad. Sci., Prague). *Coll. Czech. Chem. Commun.*, 1960, **25** (12), 3071–3084 (in German).—Oscillographic polarography may be used successfully for the examination of non-reducible substances which are adsorbed on the mercury electrode and alter the capacity of the electrical double layer. The effect appears on the curve $dE/dt = f(E)$ as an incision of adsorption and desorption. The capacity effect is related to the solubility of the substance in the supporting electrolyte; the lower the solubility the deeper the incision. Thus a substance such as ethanol, which is completely miscible with water, will show a good incision only if a supporting electrolyte is chosen in which it is less soluble, *i.e.*, 10M to 15M-NaOH. As soon as the substance begins to precipitate from the supporting electrolyte, very sharp incisions appear, indicating the formation of a complex film. Analytical determinations may be carried out by measuring the depth of the incision or by comparing the curve with those of standards. It is possible to differentiate between alcohols and their isomers. Normal alcohols in lower concn. give the same depth of incision as do the corresponding iso-alcohols. In some cases differentiation between two members of a homologous series is possible owing to the different potentials of their incisions at the concn. at which the incisions of both substances have the same depth (*e.g.*, primary and tertiary alcohols, aromatic hydrocarbons and acids of the indole series).

XIII. Application of a semi-conductor probe for signalling the depth of incisions on oscillographic curves. R. Kalvoda. *Chem. Listy*, 1960, **54** (12), 1299–1303.—The device described enables the impact of the electron-beam on the screen of the oscillograph to be indicated optically or acoustically and was used for measurements in oscillographic polarography. It could be used, for example, in the control of the course of slow chemical reactions.

A. J. BENNETT
J. ZÝKA

3131. **New electronic device for recording oscillographic polarograms by means of a single alternating-current cycle.** L. Molnár and E. Biró (Dept. Nat. Chem. Inst., Acad. Sci., Bratislava, Czechoslovakia). *Chem. Zvesti*, 1960, **14** (11–12), 849–858.—The device consists of two main parts—a current synchroniser and amplifier. These enable so-called "first curves" to be studied, either over the whole range or in the cathodic or anodic region, when using the $dE/dt = f(E)$ function. J. ZÝKA

3132. **Triangular potential impulse polarography.** P. Valenta and J. Volke (Polarographic Inst., Acad. Sci., Prague). *Chem. Listy*, 1960, **54** (12), 1279–1298.—Principles and possibilities of the use of $i = f(E)$ curves in the study of electrode processes and in qualitative and quantitative analysis are discussed. (31 references.) J. ZÝKA

3133. **The graphite electrode. An improved technique for voltammetry and chronopotentiometry.** P. J. Elving and D. L. Smith (Univ. of Michigan, Ann Arbor, U.S.A.). *Anal. Chem.*, 1960, **32** (13), 1849–1854.—*Cf. U.S. Atomic Energy Comm.*, Rep. TID-6167, 1960; *Anal. Abstr.*, 1961, **8**, 2255.

3134. **Automatic polarisation-potential titration with alternating current.** P. Dickens and P. König (Mannesmann A.-G., Hüttenwerk Huckingen, Duisburg, Germany). *Arch. Eisenhüttenw.*, 1960, **31** (10), 603–606.—In the titration of Ca and Mg with EDTA, the end-point is the change in the potential drop (E) between two platinum electrodes immersed in the solution (*cf.* Abresch and Büchel, *Anal.*

Abstr., 1961, **8**, 3135) and fed with a.c. at 2 to 6 V at 50 c/s. *E* is balanced bridge-wise in a double-triode valve against an adjustable reference voltage. The bridge output is amplified and rectified, and may be read as an end-point indicator, but is preferably fed to an electronic relay which closes the burette tap when the end-point is reached (*cf.* Mullen and Lingane, *Anal. Chem.*, 1948, **20**, 795). Details of the application to ores, slag, dolomite and refractories are given. The method is also applicable to redox and pptn. titrations. A. R. PEARSON

3135. **Application of polarisation-potential titration in steel-works laboratories.** K. Abresch and E. Büchel (A. Thyssen-Hütte A.-G., Duisburg-Hamborn, Germany). *Arch. Eisenhüttenw.*, 1960, **31** (10), 595–601; 606.—This method (*cf.* Reilley *et al.*, *Anal. Chem.*, 1951, **23**, 1223; Franck, *Anal. Abstr.*, 1955, **2**, 19; *Z. Electrochem.*, 1958, **62**, 245) differs from the usual electrometric titration in that the end-point depends, not on an electrode potential set up by the solution being titrated, but on the change in current or p.d. between two small spherical platinum electrodes, sealed $\simeq$4 mm apart in a glass tube and immersed in the soln., to which a polarising p.d. is applied. In the potentiometric procedure the choice of a suitable value for the current is important, *e.g.*, in the titration of CrO_4^{2-} with Fe^{2+} at a polarising current of $\simeq$0·25 mA, the change of p.d. between the electrodes at the end-point was $\simeq$400 mV. Vanadium in steel was similarly titrated. If both V and Cr are present they are titrated together, and V is determined separately by oxidation with $KMnO_4$ and back-titration. Applications to the determination of Pb in steel and galvanising metal, Sn in pig-iron and plain steel, and Mn in ores and ferromanganese are described. The method has also been applied to titrations of As^{3+} and Sb^{3+} with BrO_3^-, Fe^{2+} with Ce^{4+}, MnO_4^- with Fe^{2+}, iodine with $S_2O_3^{2-}$, Zn^{2+} with $K_4Fe(CN)_6$, Cu^{2+} with I^- or $S_2O_3^{2-}$, and Ag^+ with halides. Instruments are described. The end-points are so much superior that the simple potentiometric methods previously used in the Thyssen laboratories have been superseded. A. R. PEARSON

3136. **Recording conductimetric titrator.** D. W. Colvin and R. C. Propst (Savannah River Lab., E. I. du Pont de Nemours and Co., Aiken, S.C.). *Anal. Chem.*, 1960, **32** (13), 1858–1861.—The instrument described automatically records the conductance of the sample soln. as a function of the vol. of titrant added. It has a linear range up to 2·6 × 10^4 μmho in 5 overlapping steps, and conductance values as high as 3 × 10^5 μmho can be recorded. Titration curves which represent small changes in conductance (50 μmho for the 350-μmho range, 2500 μmho for the 2·6 × 10^4 μmho range) can be adjusted so as to occupy the full width of the recorder chart. D. C. ARMSWORTH

3137. **Coulometry with the carbon black electrode.** J. D. Voorhies and S. M. Davis (Org. Chem. Div., American Cyanamid Co., Bound Brook, N.J.). *Anal. Chem.*, 1960, **32** (13), 1855–1857.—A description is given of a modification of direct constant-current coulometry involving quant. adsorption of an electroactive species on to acetylene black and subsequent quant. electrolysis of the adsorbed substance. Carbon-electrode coulometry seems to have the greatest potential value in the range of semi-micro analysis of electro-active organic compounds of low solubility in H_2O. The best results are obtained by electrolysis in concentrated salt soln. D. C. ARMSWORTH

3138. **Improved α-counting technique.** N. A. Hallden and J. H. Harley (Health and Safety Lab., U.S. Atomic Energy Commission, 376 Hudson St., New York 14). *Anal. Chem.*, 1960, **32** (13), 1861–1863.—A scintillation technique for α-counting with a zinc sulphide phosphor gives higher efficiency and lower background than the standard techniques. The zinc sulphide is coated on a Mylar base and discs of the phosphor are applied directly to the sample. The efficiency is $\simeq$47%, with a background of $<$1 count per hr. The procedure has been applied to the routine α-counting of air-dust samples on filter-paper and to the measurement of Ba-$RaSO_4$ ppt. obtained in the determination of Ra in urine and water by co-pptn. with $BaSO_4$. D. C. ARMSWORTH

3139. **4-Pi zinc sulphide α-counting.** N. I. Sax and D. Rosi (Div. of Lab. and Res., New York State Dept. of Health, Albany). *Anal. Chem.*, 1960, **32** (13), 1900.—A Mylar and plastic-dispersed zinc sulphide film is used. Two pieces of plastic film are coated on the dull surface with the sample and placed face to face in the well of the scintillation spectrometer. The efficiency is >90% in 4π α-counting. The background is maintained at 0·1 count per min., thus permitting the measurement of 10^{-14} g of Ra per sample. D. C. ARMSWORTH

3140. **Simple method of absolute β-counting by scintillation technique.** M. G. Shahani (Atomic Energy Estab., Trombay, Bombay). *J. Sci. Ind. Res., India, B*, 1960, **19** (12), 461–464.—The detector consists of a cylinder of plastic phosphor (4 cm in diam. × 3·5 cm in height) cut into two hemi-cylindrical pieces; after the radioactive soln. had been dried on the rectangular face of one of the pieces they were mounted together on a photomultiplier. The total counting rate was determined by recording the number of pulses with amplitudes >6·8 V and adding to this the number of pulses with lesser amplitudes, derived by extrapolation of a graph of count rate *vs.* pulse height for the particular voltage applied to the photomultiplier. Results for ^{32}P are quoted; the total count rate was virtually constant over the range 600 to 900 V. G. J. HUNTER

See also Abstract—**2793**, A.c. polarographic circuit.

ABBREVIATIONS

Certain abbreviations in everyday use are not included in the following list. When any doubt might arise from the use of an abbreviation or symbol the word is printed in full.

alternating current	a.c.
ampere	amp.
Ångström unit	Å
anhydrous	anhyd.
approximate, -ly	approx.
aqueous	aq.
atmospher-e, -ic	atm.
boiling-point	b.p.
British thermal unit	B.Th.U.
calorie (large)	kg-cal.
calorie (small)	g-cal.
centimetre	cm
coefficient	coeff.
Colour Index	C.I.
concentrated	conc.
concentration	concn.
constant	const.
crystalline, crystallised	cryst.
cubic	cu.
current density	c.d.
cycles per second	c/s
density	ρ
density, relative	d or wt. per ml
dilute	dil.
direct current	d.c.
distilled	dist.
electromotive force	e.m.f.
equivalent	equiv.
ethylenediaminetetra-acetic acid	EDTA
gram	g
gram-molecule	mole
half-wave potential	$E_{\frac{1}{2}}$
hour	hr.
hydrygen ion exponent	pH
infra-red	i.r.
insoluble	insol.
international unit	i.u.
kilogram	kg
kilovolt	kV
kilowatt	kW
liquid	liq.
maxim-um, -a	max.
melting-point	m.p.
microgram	μg (not γ)
microlitre	μl
micromole	μmole
micron	μ
milliampere	mA
milli-equivalent	milli-equiv.
milligram	mg
millilitre	ml
millimetre	mm
millimicrogram	mμg
millimolar	mM
millivolt	mV
minute (time)	min.
molar (concentration)	M
molecul-e, -ar	mol.
normal (concentration)	N
optical rotation	α_l
ounce	oz
parts per million	p.p.m.
per cent.	%
per cent. (vol. in vol.)	% (v/v)
per cent. (wt. in vol.)	% (w/v)
per cent. (wt. in wt.)	% (w/w)
potential difference	p.d.
precipitate (as a noun)	ppt.
precipitated	pptd.
precipitating	pptg.
precipitation	pptn.
preparation	prep.
qualitative, -ly	qual.
quantitative, -ly	quant.
recrystallised	recryst.
refractive index	n_λ^t
relative band speed	R_F
relative humidity	r.h.
revolutions per minute	r.p.m.
saponification value	sap. val.
saturated calomel electrode	S.C.E.
second (time)	sec.
soluble	sol.
solution	soln.
specific gravity	sp. gr.
specific rotation	$[\alpha]_\lambda^t$
square centimetre	sq. cm
standard temp. and pressure	s.t.p.
temperature	temp.
ultra-violet	u.v.
vapour density	v.d.
vapour pressure	$v.p._t$
volt	V
volume	vol.
watt	W
wavelength	λ
weight	wt.

In addition, the following symbols may be used in conjunction with numerical values or in mathematical expressions—

greater than	$>$	less than	$<$
not greater than	$\ngtr$	not less than	$\nless$
is proportional to	$\propto$	of the order of, approximately	$\simeq$

The principal Pharmacopoeias are denoted by B.P., U.S.P. or D.A.B., together with the identifying roman numeral or year.

Valency states are represented by a superscript roman numeral, *e.g.*, Fe^{II}, Mo^{V}. Substances in the ionic state are represented by Na^+, Fe^{3+}, Fe^{3+}, etc., for cations and by Cl^-, SO_4^{2-}, PO_4^{3-}, etc., for anions.

The chemical nomenclature used in the abstracts is that recommended by The Chemical Society ("Handbook for Chemical Society Authors", The Chemical Society, London, 1960), and may differ from that used in the original articles.

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